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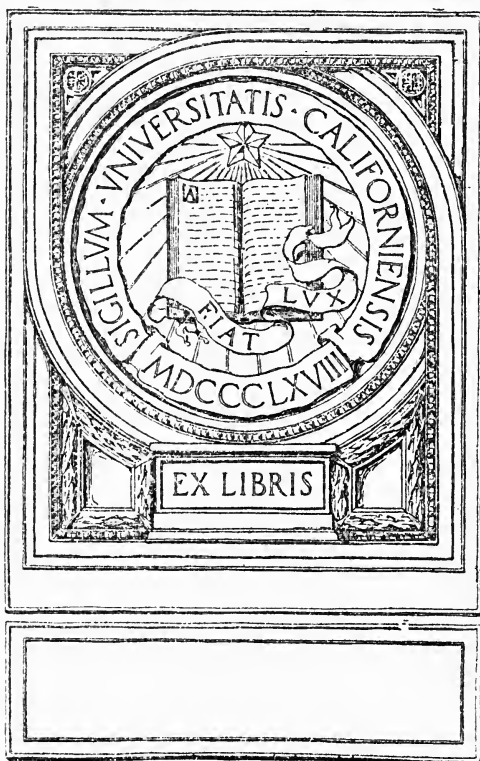
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LABORATORY DIRECTIONS
FOR
ELEMENTARY CHEMISTRY
MATTILL

Edmund O'Neill

IN MEMORIAM

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LABORATORY DIRECTIONS FOR ELEMENTARY CHEMISTRY

DESIGNED TO ACCOMPANY
A TEXTBOOK OF CHEMISTRY
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IN MEMORIAM

TO THE
MEMORIAL

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PREFACE

The importance of the laboratory work in a course in elementary chemistry cannot be too strongly emphasized. It is only by the actual contact and experimentation with some of the many materials described in a text-book that the student ever comes to any realizing sense of the means by which the science of chemistry has been built up. For this reason it is advisable to supplement the laboratory exercises with discussions of an explanatory nature, in which the correlation between laboratory and text-book work may be brought out.

The experiments described in this guide have been chosen with the following objects in mind: To reduce the variety of materials handled, consequently the confusion of many new names and strange materials, to a minimum; to make each experiment a fairly exhaustive study of some particular material or property; to have the sequence of experiments such as will lead to a certain amount of reasoning from analogy. This guide does not pretend to be an exhaustive description of elementary experiments, but rather a selection of a few suitable, consecutive experiments from the many possible.

I wish to take this opportunity to acknowledge the many helpful suggestions and criticisms which I have received from Drs. C. W. Balke, C. H. Hecker, S. B. Hopkins and W. A. Noyes in the preparation of this book.

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LABORATORY DIRECTIONS FOR ELEMENTARY CHEMISTRY

GENERAL DIRECTIONS

Work in the laboratory should be undertaken with the following objects in mind: 1. By direct handling of the materials to become familiar with certain chemical reactions. 2. By analogy, and text-book study, to relate the experiments actually performed in the laboratory with as many facts brought up in the lecture and quiz room discussion as possible. 3. By handling and manipulating apparatus to become familiar with the tools at the disposal of the chemist, and the proper use of the same, and at the same time to acquire ability and dexterity in their use. A student, however good his understanding of chemical facts, has not reached the goal unless he is able to perform neatly and properly any given chemical manipulation. 4. To acquire habits of observation and a proper discrimination between important and unimportant details, and to draw conclusions of a general nature from a specific case. 5. To record accurately, briefly, and promptly the phenomena observed.

In the pursuit of these objects the keeping of a laboratory note-book is of prime importance. It is absolutely essential that these notes be recorded at the time the work is performed. They should give a clear but brief account of work done and should include a statement of the object of the experiment, a record of all phenomena observed, such as change of color, appearance of a precipitate or a gas, etc., and answer all ques-

tions. A drawing of the apparatus used may often reduce the length of the description required. Wherever possible a chemical equation should be written to express the change taking place. In order to write such equations it may be necessary to refer to the text-book for the products of the reaction. After the products have been determined the equation should be written and balanced without reference to text-book or other aid. *Do not delay in recording the observations made during a given experiment; each change should be jotted down as observed.* It is more essential that the note-book be a record of the experiment taken at the time it is performed than that it should be neat and well written, though both of these ends may be accomplished at once without too great difficulty. Under no circumstances should the record of an experiment be placed on loose paper, the loss of which would mean the repetition of the experiment so recorded.

The following general directions for manipulation should be closely followed:

(1) Do not use too much material. The amount called for in any experiment is ample to secure the results desired. Taking larger amounts of material is wasteful, frequently dangerous, and always results in loss of time.

(2) Keep all bottles in their proper places.

(3) Material spilled on the side shelf should be promptly swept into a waste jar.

(4) Never place the stopper of a reagent bottle on the table—hold it between the fingers.

(5) Do not dip stirring rods or tubes into the reagent bottles. Pour a little of the reagent into a test tube.

(6) Do not waste gas or water.

(7) In pushing a glass tube through a stopper wet both the tube and the stopper. It is wise also to protect the hand by covering the tube with a towel.

(8) Do not heat beakers or flasks with the direct flame, but always place them on a wire gauze. Test tubes are the only glass receptacles which can be heated in the direct flame.

(9) Before leaving at the end of a laboratory period see that all apparatus is in its proper place, and that the desk is clean.

DIRECTIONS FOR QUANTITATIVE EXPERIMENTS

In performing quantitative experiments the student must keep in mind the object of the given experiment, and the fact that successful results in quantitative work depend largely on the care and accuracy with which given manipulations are carried out. A given experiment may be successful in the hands of one student and a complete failure in the hands of another, not because of any fault in the choice of the experiment, but because of the differences in manipulation which result from an incomplete understanding of the object of the experiment, and of the possibilities of error and how to avoid them. For this reason it is especially essential that the directions for an experiment marked *quantitative* should be studied from beginning to end before any manipulation is started. A student should begin the manipulation of a quantitative experiment with the object of the experiment so clearly in mind that reference to the directions is unnecessary except as regards the details of manipulation.

The record of a quantitative experiment should be kept in tabulated form, and should in every case be kept *in the note-book*. *If the experiment has to be repeated the record of every succeeding trial must also be in the note-book.* On reading over the directions make a note of what measurements are to be taken and in what order they will be used, then prepare a sheet of the laboratory note-book with the proper headings in the proper order before starting the experiment. For example, in the first experiment (No. 8) the weights of two tubes empty, with potassium chlorate, and with the residue on heating potassium chlorate are to be determined and from that data the percentage of potassium chloride obtained on heating a given amount of potassium chlorate is to be calculated. The headings then might be arranged as follows:

	Small tube	Large tube
Tube + potassium chlorate,
Tube empty,
Potassium chlorate,
Tube + potassium chloride (residue),
Tube empty,
Potassium chloride,
Per cent of potassium chloride in potassium chlorate,

The arrangement of the data sheet before performing the experiment is a good test of the student's understanding of the object of the experiment, and for that reason should be recorded in the note-book before the manipulation is begun.

While the directions for each quantitative experiment describe the special precautions to be observed in the given experiment there are other precautions which apply to all quantitative work and which should be kept constantly in mind, of which the following are the most important.

Weighing.—In all quantitative experiments use the chemical balance. To prepare a dish for weighing it should be carefully cleaned and wiped dry with a clean towel. After a dish has been prepared for weighing it should be handled as little as possible with the fingers because of deposition of moisture and oil from the skin. Material may be placed in a dish so prepared and the required processes carried out according to the directions of the given experiment, but the student should always keep in mind the fact that an error may be introduced into the weight of any given material by allowing foreign matter to fall by accident into the dish, or by accidental loss of material from the dish. Directions to avoid loss of material by spattering, etc., must be carefully observed. The success of every quantitative experiment depends on determining by weighing, measuring or otherwise, *all* of a given material which results from a given chemical process.

Testing for Leaks.—Some of the quantitative experiments involve the evolution and measurement of a volume of gas.

In all such experiments the apparatus must be constructed with unusual care and tested to determine that there is no possibility of a loss of gas through leaks. Rubber, instead of cork stoppers should be used and wherever rubber tubing is used in making connections the glass tubes, which should always be fire polished (see Exp. 3 *a*), should meet inside the rubber tubing. *Unless an apparatus is air-tight it should not be used in a quantitative experiment.*

STANDARD CONDITIONS FOR GASES

In order that gas volumes may be comparable they must be given under fixed conditions, since the volume of a given sample of gas varies with both the temperature and the pressure under which it exists. The fixed conditions which have been arbitrarily chosen by chemists are that the gas should be dry, under the pressure of 760 mm. of mercury measured at 0° C., and at a temperature of 0° C. The gas as obtained in the laboratory is usually saturated with moisture, though in certain cases, for example Exp. 20, it may be measured dry, and is under the temperature and pressure conditions obtaining in the laboratory. These conditions may be determined by a thermometer and barometer. The barometer reading may indicate a certain atmospheric pressure, but it is self-evident that if the temperature of the room were lower the same atmospheric pressure would give a lower barometric reading, because of the contraction of the mercury on cooling. For this reason the barometric reading must be corrected for the temperature at which the barometer stands. A table of corrections for ordinary temperatures can be readily prepared from the coefficient of expansion of mercury, and may be found below. To correct a barometric reading to 0° C., then, the observed reading should be reduced by the amount indicated in the table, for the observed temperature. For instance suppose the barometer reads 74.3 cm. at 17° C., the correction for 17° C. is 2.2 mm., therefore the barometric reading at 0° C. would be $743 - 2.2 =$

740.8 mm. But if the gas was saturated with moisture the pressure indicated by the barometer was being divided between the two components of the mixture, the water vapor and the gas. Since the pressure of water vapor at varying temperatures has been accurately determined, that may be obtained from a table, and the pressure of the gas in the mixture is then the difference between the barometric reading and the pressure of water vapor at the observed temperature. For instance, the corrected barometric reading, as found above, was 740.8 mm., but the gas in question was saturated with moisture. The vapor pressure of water at 17° C. is found from the table to be 14.5 mm., therefore the pressure of the dry gas was $740.8 - 14.5 = 726.3$ mm. of mercury measured at 0° C. The gas volume must then be reduced to standard conditions of temperature and pressure by applying the laws of Boyle and Charles, before any comparison of volume may be made.

TABLE I

TEMPERATURE CORRECTIONS FOR REDUCING A COLUMN OF MERCURY 750 MM.
HIGH WHEN READ ON A GLASS SCALE, TO 0° C., IN MILLIMETERS

Temperature,	15	16	17	18	19	20	21	22	23	24	25
Correction,	2	2.1	2.2	2.3	2.5	2.6	2.7	2.8	3.0	3.1	3.2
Temperature,	26	27	28	29	30	31	32	33	34	35	
Correction,	3.4	3.5	3.6	3.7	3.9	4.0	4.1	4.3	4.4	4.5	

TABLE II

VAPOR PRESSURE OF WATER IN MM. OF MERCURY

Temperature,	0°	5	10	15	16	17	18	19
Pressure,	4.6	6.5	9.2	12.8	13.6	14.5	15.5	16.5
Temperatures,	20	21	22	23	24	25	26	27
Pressure,	17.5	18.7	19.8	21.1	22.4	23.8	25.2	26.7
Temperature,	28	29	30	31	32	33	34	35
Pressure,	28.4	30.1	31.8	33.7	35.7	37.7	39.9	42.2
Temperature,	40	45	50	55	60	65	70	75
Pressure,	55.3	71.9	92.5	118.1	149.5	187.6	233.8	289.3
Temperature,	80	85	90	95	100			
Pressure,	355.5	433.8	526.0	634.0	760			

TABLE III

DENSITIES AND PER CENT COMPOSITION OF WATER SOLUTIONS OF ACIDS
AND AMMONIA. (Taken from Landolt, Börnstein and Roth)

Acetic acid		Nitric acid		Phosphoric acid	
Sp. Gr.	% acid	Sp. Gr.	% acid	Sp. Gr.	% acid
$= d_4^{20}$		$= d_4^{15}$		$= d_4^{17.5}$	
1.012	10	1.06	10.67	1.06	10
1.026	20	1.12	20.22	1.12	20
1.038	30	1.19	30.87	1.18	30
1.049	40	1.25	39.8	1.26	40
1.057	50	1.31	49.05	1.34	50
1.064	60	1.37	59.36	1.43	60
1.069	70	1.39	63.2	1.53	70
1.070	80	1.42	69.77	1.65	80
1.066	90			1.76	90
1.050	100				

Maximum density is at
78% acid.

Hydrochloric acid		Sulfuric acid		Ammonia	
Sp. Gr.	% acid	Sp. Gr.	% acid	Sp. Gr.	%NH ₃
$= d_4^{15}$		$= d_4^{15}$		$= d_4^{15}$	
1.05	10.17	1.068	10	0.96	10
1.10	20.00	1.14	20	0.92	20
1.15	29.57	1.22	30	0.90	28.5
1.16	31.52	1.30	40		
1.20	39.11	1.40	50		
		1.50	60		
		1.61	70		
		1.73	80		
		1.82	90		
		1.84	100		

TABLE IV

SOLUBILITIES. (Taken from Landolt, Börnstein and Roth.) Giving in order the formula of the salt or its hydrate stable at 10°, the weight in grams of the salt, or the hydrate (always calculated from the formula given in column 1) required to make a liter of normal solution, and the weight of anhydrous substance in 100 grams of a saturated solution at 10°, 20°, 30°, 40°, 50° and 100°.

Formula	Grams per liter for a normal solution ¹	Solubility					
		10°	20°	30°	40°	50°	100°
AgC ₂ H ₃ O ₂	166.9	0.875	1.037	1.215	1.413	1.637
AgCl.....	143.3	8.9·10 ⁻⁵	1.53·10 ⁻⁴	2.19·10 ⁻³
AgBr.....	187.8	0.84·10 ⁻⁵	3.7·10 ⁻⁴
AgI.....	234.8	3.53·10 ⁻⁷
AgNO ₃	169.9	61.5	68.3	73.	77.	80.	90.1
Ag ₂ SO ₄	155.9	0.78	1.46
AlCl ₃ ·6H ₂ O.....	241.45
Al ₂ (SO ₄) ₃ ·18H ₂ O.....	111.1	25.1	26.6	28.8	31.4	34.3	47.1
Al ₂ K ₂ (SO ₄) ₄ ·24H ₂ O.....	118.7	7.74	60.6 xaq.
BaCO ₃	98.7	1.8·10 ⁻³
BaCl ₂ ·2H ₂ O.....	140.2	25.	26.3	27.6	29.	30.4	37.
BaO·9H ₂ O.....	157.7	2.17	3.36	4.75	6.85	10.5
CaCO ₃	50.	2·10 ⁻³
CaCl ₂ ·6H ₂ O.....	109.5	39.4	42.7	53.5 4aq	61.4 2aq
CaO·H ₂ O.....	37.	0.123	0.113	0.104	0.096	0.05
CaSO ₄ ·2H ₂ O.....	86.	0.192	0.20	0.21	0.21
CdCl ₂ ·2½H ₂ O.....	114.1	47.4	56.3	57.5 1aq	59.5
Cd(NO ₃) ₂ ·4H ₂ O.....	154.3	58.4	61.4
CoCl ₂ ·6H ₂ O.....	119.	31.	33.3	36.1	39.4	48.3	50.7 2aq
Co(NO ₃) ₂ ·6H ₂ O.....	145.6
CrO ₃	50.	62.5	64.6	67.4
Cu(NO ₃) ₂ ·6H ₂ O.....	147.9	55.6	61.5 3aq
FeCl ₃ ·6H ₂ O {	90.	45.	47.9	51.6
FeSO ₄ ·7H ₂ O.....	139.	17.	21.	24.8	28.7	32.7	34.2 an ³
H ₃ BO ₃	21.	4.9	6.4	8.	10.3	28.2
HgCl.....	236.	0.38·10 ⁻⁴	7·10 ⁻⁴
HgCl ₂	135.6	6.19	6.9	8.7	10.2	35.
KBr.....	119.	38.	39.4	43.2	51.2
KBrO ₃	167.	6.54	11.7	33.2
K ₂ CO ₃ ·2H ₂ O.....	87.	53.3	53.9	54.8	60.9
KCl.....	74.6	23.8	25.5	27.2	28.7	30.	36.
KClO ₃	122.6	4.7	6.8	9.2	12.7	16.5	35.9
KClO ₄	138.6	1.9 (25°)	5.	15.8
K ₂ CrO ₄	97.2 (64.8) ⁴	37.9	38.6	39.5	40.1	40.8	44.2
K ₂ Cr ₂ O ₇	147. (49) ⁴	7.8	11.6	15.4	22.6	50.5
KHCO ₃	50. ²	21.7	24.9	28.1	31.2	34.2
KI.....	166.	57.7	59.1	60.4	61.5	62.7	67.6
KIO ₃	214.	7.5	10.5	11.4	24.4
KMnO ₄	158. (31.6) ⁴	4.01	6.	8.3	10.4	14.3
KNO ₃	101.	17.7	24.1	31.6	39.2	46.3	71.1
K ₂ SO ₄	87.2	8.4	10.	11.5	13.	14.	19.4
MgCl ₂ ·6H ₂ O.....	101.7	34.9	35.3	36.5	42.2
Mg(NO ₃) ₂ ·6H ₂ O.....	128.3	42.	45.9
MgSO ₄ ·7H ₂ O.....	123.3	23.6	26.2	29.	31.	33.5 6aq	42.5
MnCl ₂ ·4H ₂ O.....	99.	44.7	49.5	53.7 2aq
MnSO ₄ ·5H ₂ O.....	120.6	38.6	39.4 1aq	37.3	24.9
NH ₄ Cl.....	53.5	25.	27.1	29.3	31.4	33.5	43.6
NH ₄ HCO ₃	39.6 ²	13.7	17.4	21.3
NH ₄ NO ₃	80.1	70.8	74.8	78.5	89.7
(NH ₄) ₂ SO ₄	66.1	42.2	43.	43.8	44.8	45.8	50.8
Na ₂ B ₄ O ₇ ·10H ₂ O.....	191. ²	1.58	3.7	9.5	35.5 aq
NaBr·2H ₂ O.....	139.	47.5	51.4	53.7	55. an
NaBrO ₃	151.	27.7	33.4	47.6

TABLE IV.—*Continued*

Formula	Grams per liter for a normal solution	Solubility					
		10°	20°	30°	40°	50°	100°
Na ₂ CO ₃ ·10H ₂ O...	143.	11.2	17.6	29.	33.2 1aq	32.2	31.
NaCl.....	58.5	26.3	26.4	26.5	26.7	26.8	28.1
NaClO ₃	106.5	49.7	56.5	67.1
Na ₂ CrO ₄ ·10H ₂ O...	171.2	33.4	44.	47. 4aq	49.	51.	55.8 an
Na ₂ Cr ₂ O ₇ ·2H ₂ O...	149.1	63.	64.	66.3	68.8	71.3	82. an
NaHCO ₃	42.2	7.6	8.8	10.	11.3	12.7
NaNO ₃	85.	44.6	46.8	49.	51.	53.	64.4
Na ₂ SO ₃ ·7H ₂ O...	126.	16.6	20.5	24.8 an
Na ₂ SO ₄ ·10H ₂ O...	161.2	8.3	16.	29.	32.5 an	31.8	29.9
Na ₂ S ₂ O ₃ ·5H ₂ O...	124.	38.	41.	46.	50.6	63. 2aq	72.7
Ni(NO ₃) ₂ ·6H ₂ O...	145.5	49.	55.
PbCl ₂	138.9	0.96	3.2
Pb(NO ₃) ₂	166.	30.8	34.	38.	41.	44.	56.
SrCO ₃	73.8	1·10 ⁻³
SrCl ₂ ·6H ₂ O.....	133.3	32.6	35.	37.5	40.	42.7	50.5
Sr(NO ₃) ₂ ·4H ₂ O....	141.8	35.5	41.5	46.7	47.7 an	48.1	50.3
SrO·9H ₂ O.....	132.8	0.48	0.7	1.	1.5	2.1	19.5
SrSO ₄	91.8	9.9·10 ⁻³	15·10 ⁻³	16·10 ⁻³	18·10 ⁻³
ZnCl ₂ ·2 1/2 H ₂ O...	91.1	73.1	78.6 1½aq	82. an	86.
Zn(NO ₃) ₂ ·6H ₂ O....	148.8	54.	67.4 3aq
ZnSO ₄ ·7H ₂ O.....	143.8	32.3	41.2	43.5 6aq	44. 1aq

¹ In many cases this quantity exceeds the solubility of the salt, but it is always given because of the chemical significance of the value.

² This is half the formula weight.

³ Anhydrous.

⁴ The figure in parenthesis applies when the salt is to be used as an oxidizing agent in acid solution.

TABLE V

SOLUBILITY AND DENSITY OF GASES. (Taken from Landolt, Börnstein and Roth.) Giving the volume of gas, measured at standard conditions, absorbed by one volume of water, at 0°, 20°, 50° and 100° and the weight per liter of the gas under standard conditions.

Gas	0°	20°	50°	100°	Weight per liter
H ₂	0.0215	0.0184	0.0161	0.0160	0.0898
N ₂	0.0239	0.0164	0.0106	0.0100	1.256
O ₂	0.0489	0.0310	0.0209	0.0170	1.429
Cl ₂	4.61	2.26	1.2	0.000	3.180
HCl	506.9	442.3	361.	1.610
Br ₂	60.5	21.3	6.5	7.143
HBr	612.5	468.	3.616
NH ₃	1305	715.4	0.762
NO	0.0738	0.047	0.0315	0.0262	1.340
N ₂ O	0.629	1.970
CO ₂	1.713	0.878	0.436	1.977
SO ₂	79.8	39.4	2.870
H ₂ S	4.6	2.5	1.4	0.8	1.542
CH ₄	0.0556	0.033	0.0213	0.017	0.717
C ₂ H ₄	0.226	0.122	1.252
C ₂ H ₂	1.73	1.03	1.162

CHAPTER I

APPARATUS AND MANIPULATION

Check up the apparatus found in the desk, using a list obtained from the storeroom. Record any missing articles on a sheet furnished for that purpose, and after getting the approval of an instructor take both lists to the storeroom and obtain the missing articles.

Study the apparatus supplied with a view to determining its uses and proper manipulation as follows:

1. Bunsen Burner.

a. Examine the construction of the Bunsen burner. Explain the function of each part.

b. Attach the burner to the gas supply by means of a rubber tube, open the holes at the bottom of the vertical tube and light the gas. What is the character of the flame? Show the structure of the flame by a sketch. Explore the various parts of the flame with a platinum wire. What are the relative temperatures of the different parts? Where should an object be placed to secure the maximum heating effect? Could a flame of this character be used for illuminating purposes? What is the principle of the Welsbach light?

c. Close the holes at the bottom of the tube. What is the character of the flame now? Hold a cold porcelain dish in the flame for a moment. What is the deposit? (Illuminating gas contains a compound of carbon and hydrogen.) Why is it possible to obtain a deposit of this substance on a cold dish? Hold a clean portion of the dish above, but not in, the flame. Why is no deposit obtained in this case? What causes the luminosity of the flame?

d. Repeat *c* using a non-luminous flame. Explain the result.

e. Extinguish the flame, turn on the gas without lighting it, and hold the dish in the current of gas. Why is no deposit obtained?

f. Light the gas and show the structure of the luminous flame by a sketch (turn the flame low to avoid flickering). Test the temperature of this flame with a platinum wire. How does it compare with the temperature of the non-luminous flame?

g. Quickly introduce the head of a match into the innermost cone of both flames. What does this experiment indicate as to

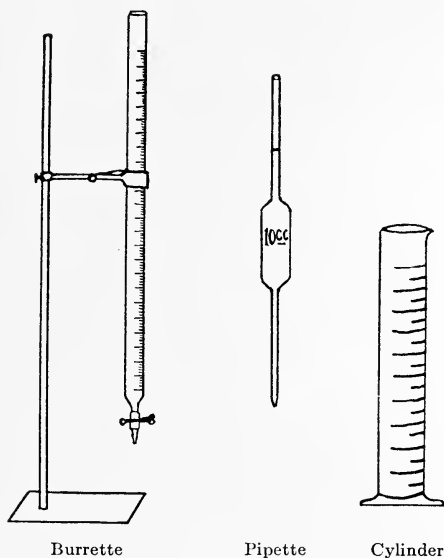


FIG. 1.

the temperature of the central cone? What composes this central portion? Devise and perform an experiment to test this point.

2. Measuring Instruments.

The measuring instruments consist of the cylinder, pipette and burette (side shelf). Fig. 1. The cylinder may be filled to the zero mark, then the amount removed read on the scale. The

pipette is used to measure one stated quantity. When the drawn out tip is placed under the surface of the liquid and suction is applied to the other end until the bottom of the meniscus of the liquid is brought to the level of the mark on the neck, the pipette will deliver the stated quantity.

The burette is filled to the zero mark, taking care that no bubbles of air remain in the parts about the nozzle, the desired quantity of liquid withdrawn from the nozzle at the bottom, and the volume withdrawn read on the scale. All readings should be taken from the bottom of the meniscus.

Fill a test-tube with 20 c.c. of water from the pipette, mark the level of the water on the tube with a paper label or file mark. Determine the capacity of the tube up to the given mark by means of the cylinder and the burette. Which gives the most accurate measure of volume, and why?

3. Working with Glass Tubing.

a. To cut glass tubing make a file mark at the desired spot, then grasping the tube with both hands place the two thumb nails directly behind the mark and break by pressing out and pulling at the same time. The sharp edges of the freshly broken glass *must* be rounded off by holding it in the hottest part (Exp. 1) of the Bunsen flame till it begins to glow, then allowing it to cool slowly. This is called fire polishing.

b. To make a bend in glass tubing it is desirable to heat the tubing uniformly through a distance of 5 to 8 cm. (2 to 3 inches), so as to avoid too great thickening of the glass at the bend. This is accomplished by holding the glass parallel with the flame of a fish tail burner, and just above the inner cone, *i.e.*, in the hottest part of the flame. The tubing should be slowly rotated in the flame *until it has softened*, then withdrawn from the flame, bent to the desired angle, and allowed to cool slowly. Fig. 2.

c. To draw out a fine capillary, or a jet, the glass should be softened for only 1 to 2 cm. of its length. What flame is best

adapted to this purpose? Rotate the glass tube in the flame until softened uniformly, then remove it from the flame and pull it out to the desired length.

d. After a little practice in making bends and jets make a wash bottle as shown in Fig. 3. A two-hole rubber stopper or a cork stopper may be used. If a cork is used it should be softened by means of a press or cork roller and the holes must

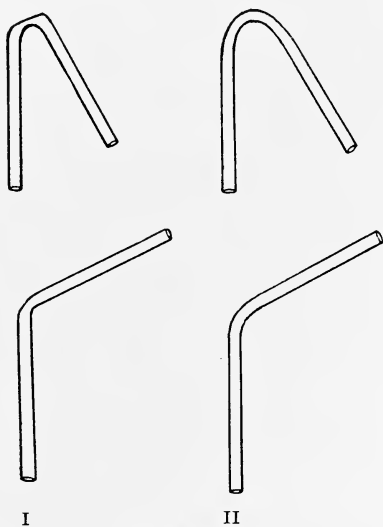


FIG. 2.

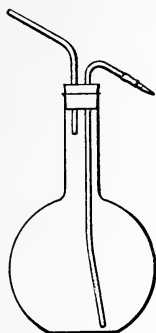


FIG. 3.

I. Shows bends made by heating the glass over a very short distance, as would be done by a Bunsen burner. The glass buckles and is uneven in thickness. II. Shows similar bends made by heating the glass over a greater distance, using a fish-tail burner. The glass is even in thickness.

be made by cutting through with a cork borer which is slightly smaller in diameter than the glass tubing. Give the cork and borer opposite rotatory motion, with a steady pressure not great enough to tear the cork, until it is cut half way through, then remove and start from the other end, placing the borer so that the cuts will meet in the middle. *Always remove the cork from inside the borer.* If the hole in the cork is too small for the glass tubing it may be enlarged by using a rat-tail file.

4. The Balance.

a. Under the direction of an instructor examine the chemical balance. Sit directly in front of the center of the balance and release the stop with a slow, steady movement. If the pointer does not swing raise the stop and release again. The swing of the pointer should be entirely within the graduated portion of the ivory scale. When the pointer is swinging freely, find the center of the arc described in its swing. This is the true zero and its position must be determined for each weighing, since the point changes.

b. Place on the left hand pan of the balance (see rules in paragraph *c*) one of the hard glass tubes which has been previously cleaned and dried (see page 4). With the aid of the forceps place on the right hand pan that combination of weights which is nearest to, but less than, the weight of the tube. Always begin with the largest weight and work down. When the tube is nearly counterpoised close the balance case and adjust the rider upon the scale beam until the pointer oscillates about the true zero, as determined in *a*. Find the weight of the tube first by adding together the values of the weights which have been removed from the set; and second by adding the weights themselves. If these two weights are identical, record in the note-book. Notice the decimal system in the weights. For example if a dish is balanced when the opposite pan contains 10 grams, 2 grams, 500 mg., 200 mg., 100 mg., 10 mg., and the rider is upon 2 on the beam, the weight of the dish is 12.812 grams. Now remove the weights, replacing each in its proper position in the set, raise the rider from the beam, and close the balance case.

c. Always observe the following rules in weighing:

- (1) Handle the balance with care.
- (2) Raise the stop gently, and always as the pointer is passing the zero mark.
- (3) Never add anything to the pans, or remove anything from them, while they are swinging.
- (4) Never place chemicals directly upon the pan. Use a

piece of glazed paper or a watch glass which has been properly counterpoised.

(5) Always use forceps in handling the weights.

(6) Place the object to be weighed and the weights as near the center of the pan as possible.

(7) Do not allow the pans to swing.

(8) In case any solid is spilled in the balance case use the camels hair brush to remove it. Keep the balance and the shelf clean.

(9) Keep both eyes open while reading the swing of the pointer.

(10) If the balance is not in proper adjustment, call an instructor.

CHAPTER II

PURE SUBSTANCES AND MIXTURES

5. Boiling Point as a Test for Purity.

This test depends on the fact that a pure substance has a fixed boiling-point, while a mixture of two or more substances has a boiling-point which changes as the boiling proceeds. In such a case the substance which has the lower boiling-point boils off in greater proportion at first, and if the vapors are con-

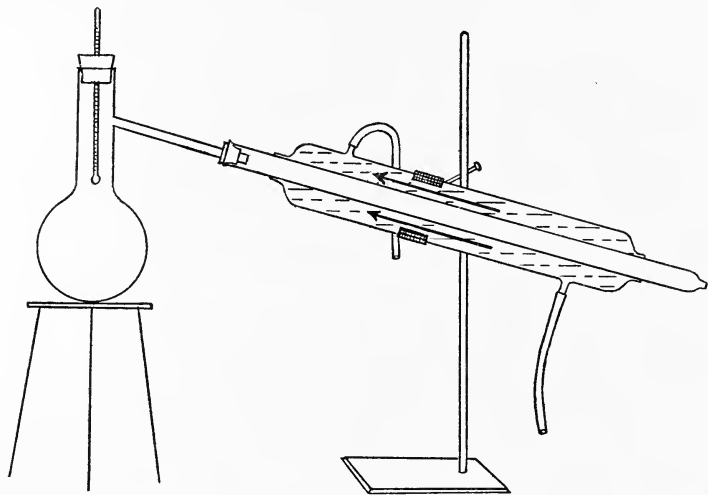


FIG. 4.

densed a liquid can be obtained which contains a higher percentage of the low boiling substance than the original mixture. This fact is utilized in many commercial processes, as in the manufacture of alcohol.

a. Connect a 50 c.c. distilling flask with a Liebig condenser. Fig. 4. Force a slow stream of cold water through the jacket

of the condenser in the direction of the arrows. Why is the water circulated in this direction? Half fill the flask with distilled water and stopper with a one-hole cork carrying a thermometer. The bulb of the thermometer should be just a little below the level of the delivery tube of the flask. Why? Heat the distilling flask with a small flame so that the water distills slowly. Collect the distillate in a small flask. After a few cubic centimeters of water have been collected read the temperature. Does this stay constant as the distillation progresses? Is it the temperature given in the text-book as the boiling-point of water? If not, give the reason for the difference. What ought to be specified when a boiling-point is given?

b. Add 5 c.c. of alcohol to 20 c.c. of water and repeat Exp. *a* with this mixture. What is its boiling-point? Do alcohol and water form a mixture or a pure substance? Collect the first few cubic centimeters of the distillate in an evaporating dish and apply a flame to the liquid. In a like manner collect a few cubic centimeters after about half of the original mixture has been distilled, and also the last few cubic centimeters. Do not heat the distilling flask to dryness. Test each portion of the distillate with the flame. Account for the difference. Is there any alcohol in the last portion of the distillate? Is there any water in the first portion?

c. Dissolve a few small crystals of copper sulfate (CuSO_4) in 25 c.c. of water and distil the mixture until about 5 c.c. have been collected. What is the distillate? From what kind of impurities can water be freed by distillation? Evaporate a little of the distillate on a watch glass, holding the watch glass about 6 inches above the tip of the bunsen flame. Evaporate tap water similarly. Why is distilled water used in the laboratory?

6. Melting-point as a Test for Purity.

This test depends on the fact that a pure substance has a fixed melting-point, while a mixture of two substances begins to melt at a temperature different from that at which it is completely melted, or, in reverse order, begins to solidify at a

temperature different than that at which it is completely solidified. A familiar example is found in the melting of ice. Pure ice melts at 0°C ., and ice and water can exist together only at 0°C . If salt (an impurity) is added to water, the water begins to freeze at some temperature below 0°C ., and the mass is not completely solid (frozen) until -23°C . is reached. To illustrate this test proceed as follows:

a. Place about 2 grams of powdered, hydrated calcium chloride ($\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$) in a clean, dry test-tube and heat *very gently* (this salt has a very low melting-point) until the substance softens enough so that the bulb of a thermometer may be inserted into it. Continue warming, while stirring with the thermometer, until a clear liquid is obtained. Now let the melted mass cool gradually, while continuing the stirring, by holding the test-tube under running water, and watch the thermometer constantly. When the solid particles first begin to appear note the temperature, and continue the cooling till the contents of the tube is solid. Has the temperature changed during the process of solidification? Has the substance a fixed freezing-point? Is it a pure substance? If the first attempt is not successful this procedure may be repeated as many times as necessary.

b. While the thermometer is still in the tube warm again, this time noting the temperature at which the substance melts. (Caution: If heat is applied too rapidly the melting-point cannot be observed; the heating should be so gradual that the solid is completely melted only after several minutes.) Is the melting-point the same as the freezing-point?

c. Now add to the tube and contents a small amount of solid sodium chloride (NaCl) and proceed as above to determine the freezing-point. Has the substance a fixed freezing-point or melting-point now? Account for the difference.

7. Elements and Compounds.

a. Grind 4 grams of sulfur and 7 grams of iron filings together in a mortar. Examine the mass with a lens. Place a

small amount of the mixture in each of three test-tubes. To the first add 15 c.c. of water and shake thoroughly. To the second add 10 c.c. of dilute hydrochloric acid. To the third add 5 c.c. of carbon disulfide, shake, filter¹ (use a dry filter) the liquid into a watch crystal and allow it to evaporate spontaneously. (Carbon disulfide is inflammable. Do not bring it near a flame.) Describe what takes place in each case.

b. Heat the remainder of the original mixture in a test-tube until it begins to glow strongly. Allow the mass to cool, reduce it to a powder, and repeat the tests made in *a*. Account for the differences in the results obtained.

c. Heat a small portion of mercuric oxide in a dry, hard glass test-tube. Watch for the appearance of the evolution of a gas, and test it with a glowing splinter. Note the change in the appearance of the residue in the test-tube, and the deposit on the cool parts of the glass. Is mercuric oxide an element or compound? Why? Have you any experimental proof for saying whether the products of this decomposition are elements or compounds?

d. Heat a small portion of copper oxalate in the same way. Test the gas evolved with a glowing splinter, and note the change in appearance of a rod wet with lime water and held at the mouth of the tube. Is the same gas evolved as before? What remains in the tube? Continue the heating until the residue in the tube shows a further change. Is this change accompanied by the evolution of a gas?

e. Define an element, and a compound. Show which materials used above have been proved to be compounds. If the weight of the residue from the copper oxalate had been determined before and after the final change from the dark red to the black substance, the change could have been shown to be accom-

¹ To prepare a filter paper fold it through the middle, then fold the straight edge onto itself, pinching the cone so made together just at the fold. Now open the cone so that there are three thicknesses of paper on one side and one on the other; fit into the funnel, taking care that the paper lies against the glass wall on all sides, finish creasing the second fold in the paper, and moisten so that it will stick in place.

panied by increase in weight. Is it then a decomposition or a combination?

8. Law of Definite Proportion. Quantitative.

Read the Directions for Quantitative Experiments (page 3) before proceeding with this experiment.

a. Weigh accurately the two hard glass ignition tubes or use those weighed in Exp. 4. In the smaller place about 0.5 gram, in the larger about 1 gram of pure, dry potassium chlorate (KClO_3) (balance room) and weigh again. The difference between the two weights is the exact weight of the potassium chlorate. Heat each tube gently in the Bunsen flame. The potassium chlorate will melt, and then appear to boil. During the boiling the heating should be just sufficient to sustain boiling, as too great heat will cause the loss of solid particles along with the escaping gas. A glowing splinter should be applied to the gas escaping from the tube, the result to be explained later. After the boiling has ceased the tubes should be heated with the hottest part of the Bunsen flame till the material is completely melted, then allow them to cool and weigh again. Repeat the heating and weighing until constant weight is obtained. The difference between the last weight and that of the empty tube gives the weight of the solid residue resulting from the decomposition of potassium chlorate by heat. *Keep these tubes with the residues until your results have been approved by an instructor.*

b. Calculate the per cent of residue in each case and compare. State the law of definite proportion and show how this experiment supports the law.

c. This example is a case of the decomposition of a more complex compound (potassium chlorate) into an element (oxygen) and a simpler compound (potassium chloride). Write the equation. How was the oxygen recognized? Dissolve the residue, *after the results of the quantitative work have been approved*, and test the solution with a solution of silver nitrate (AgNO_3). Apply the same test to a solution of some of the original potassium chlorate. Interpret the results.

CHAPTER III

OXYGEN

9. Catalysis.

a. Melt a small sample of potassium chlorate (KClO_3) in a dry test-tube and note the rate at which decomposition proceeds; then drop into the melted mass a pinch of manganese dioxide (MnO_2) (known to be free from organic matter) and note the change in the rate of decomposition. Allow the mass to cool and dissolve. Has there been any apparent change in the manganese dioxide?

b. Place as much sodium peroxide (Na_2O_2) as can be piled on a dime in each of two porcelain crucibles; to one of the crucibles add a very small amount of copper oxide (CuO), then fuse the material in each crucible, allow to cool and to each add water and note the rate of evolution of gas. Test the gas evolved with a glowing splinter. What is it? Does the copper oxide have any effect on the nature of the chemical change? On the speed? Has it undergone any change during the reaction? Boil the mixture in the crucible containing the copper, and filter. The copper oxide (CuO) was converted into the hydroxide ($\text{Cu}(\text{OH})_2$ or $\text{CuO} \cdot \text{H}_2\text{O}$) by the fusion, and on boiling changed back to copper oxide.

10. Preparation and Properties of Oxygen.

a. Calculate from the data of Exp. 8, and from the capacity of a gas-collecting bottle and the weight of 1 liter of oxygen, the amount of potassium chlorate (KClO_3) required to give oxygen enough to fill four of the gas-collecting bottles. Weigh out the calculated amount of potassium chlorate on the rough laboratory balance, mix with one-fourth its volume of manganese dioxide (MnO_2) and place in the large

hard glass test-tube which should be fitted with a stopper carrying an outlet tube, and which must be proved to be air-tight before proceeding. A small portion of this mixture should be tested by heating in another tube. If the reaction takes place quietly it is safe to conclude that organic matter is absent and the reaction will proceed quietly in the large tube. Connect the delivery tube with a glass tube leading to the trough in which the gas-collecting bottles have been filled with water and inverted (Fig. 5) and gently heat the potassium chlorate till gas is evolved, collecting the gas in the inverted bottles. After the bottles have been filled with gas a glass plate should

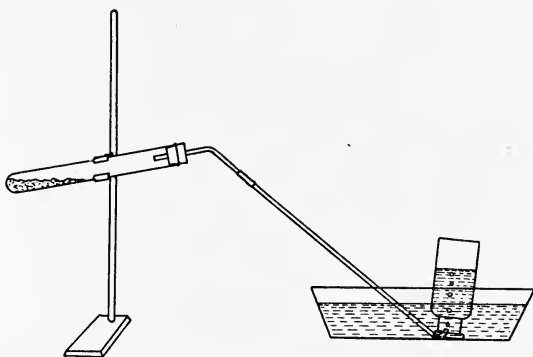


FIG. 5.

be placed over the mouth while still under water, then the bottle and cover may be removed and set aside for future use. Always withdraw the delivery tube from below the surface of the water before allowing the generating tube to cool. Why? The supply of the gas may be stopped almost instantaneously at any time by withdrawing the heat from the generating tube.

Test the four bottles of the gas as follows, covering the bottles again as soon as the spoon is withdrawn.

b. Into one insert an iron wire or picture cord, the tip of which has been heated and dipped into sulphur, then lit.

c. Into the next insert some burning sulphur on the iron spoon.

d. Into the third insert a spoon carrying some burning red phosphorus.

e. Into the fourth insert some burning magnesium ribbon, held with the pincers.

f. Add about 10 c.c. of water to each of the last three bottles and shake, then test the solutions with both blue and red litmus paper, testing at the same time a sample of the water added. What has been the nature of the reaction in these cases? Why did combustion cease before all the material had burned? Are the products of combustion soluble in water? To what can you ascribe the difference in the effect on litmus paper? Classify these three elements according to the effect of the solution of their oxides on litmus. Are they at the same time classified with respect to any physical properties? Why was it necessary to test a small portion of the mixture of potassium chlorate and manganese dioxide by heating separately? What would be the result if combustible matter, as charcoal, had been present in the mixture?

11. The Effect of Concentration on the Speed of a Reaction.

Will iron wire burn in the air as it does in oxygen? Burn sulfur, phosphorus and magnesium in bottles filled with air. Note the rate of combustion as compared with burning in oxygen and account for the difference. Add water and test with litmus as in Exp. 10 *f*. Are the products of the reaction the same in the two cases?

12. To Determine the Weight of a Liter of Oxygen. Quantitative.

Various methods for determining the density, or weight per liter of a gas, may be used. In the following experiment the weight of the gas will be determined by the loss in weight of a material, due to evolution of the gas in question, while the volume of the gas will be measured directly. Having the weight of a given volume the weight per liter can then be calculated by direct proportion.

a. Fit the large ignition tube with a rubber stopper and delivery tube bent so that it can be connected with a gas burette (Fig. 6). Fill the burette with water and see that there is no air in the connecting rubber tubing by raising and lowering the reservoir (R) several times, while the pinch cock is open. Connect the burette with the ignition tube and test for air tightness. Then remove the ignition tube, weigh it, add 0.2 to 0.5 gram of potassium perchlorate (KClO_4) and weigh again. Bring the level of the water in the burette near the zero mark, attach the

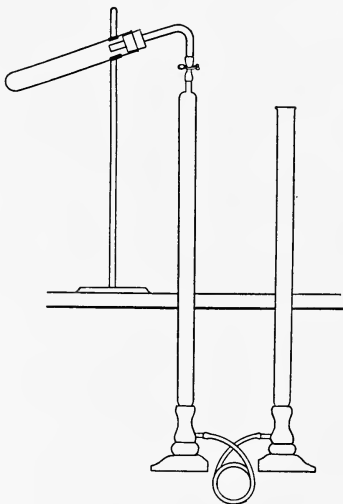


FIG. 6.

ignition tube and open the cock. If the level of the water in the burette remains constant for two minutes (showing the apparatus to be air tight) bring the gas to atmospheric pressure by raising or lowering the reservoir until the level of the water in the two tubes is the same, then record the burette reading. Now heat the potassium perchlorate *gently* until about 30 c.c. of gas has been evolved, *then allow to stand till the tube has reached room temperature*. Read the volume of the gas in the burette after bringing the level of the water in the reservoir even with that in the burette (the cock should remain open

through all this procedure), read the temperature and barometric pressure in the laboratory, then remove and weigh the ignition tube and residue. If moisture has collected in the ignition tube allow it to stand over night, or until it has completely dried by spontaneous evaporation, before weighing. The difference between the two burette readings is the volume of oxygen, the difference between the weights of the tube and potassium perchlorate and the tube and residue is the weight of the oxygen.

b. Since the volume of a gas varies greatly under varying conditions of temperature and pressure, it is convenient to state the weight of a given volume under "standard" conditions, *i.e.*, at 0° C. and 760 mm. pressure. Calculate the volume the oxygen would occupy under standard conditions, remembering that the gas measured in the burette is a mixture of oxygen and water vapor, and that the atmospheric pressure read on the barometer must be corrected for temperature (see page 5). The pressure correction to be applied for both water vapor and the temperature of the barometer may be found in tabulated form on page 6. From the weight of the given volume of oxygen calculate the weight of one liter. Calculate the per cent by which your result differs from the accepted value, 1.429 grams. *Keep the ignition tube and residue for further work.* (Exp. 24.)

c. Why is it necessary to allow for the vapor pressure in the gas? Why does not the fact that the ignition tube was full of air, some of which has gone with the oxygen into the gas burette, vitiate the results? How would it affect the result if the ignition tube had not cooled completely before reading the volume of oxygen? Why must the water in the reservoir be brought to the level of that in the burette before reading the volume of oxygen?

CHAPTER IV

HYDROGEN

13. Preparation and Properties of Hydrogen.

a. Construct an apparatus consisting of a 250 c.c. generating flask (the Erlenmeyer is well adapted to this purpose) stoppered with a cork carrying a thistle tube reaching to within 0.5 cm. of the bottom of the flask and a delivery tube bent for the

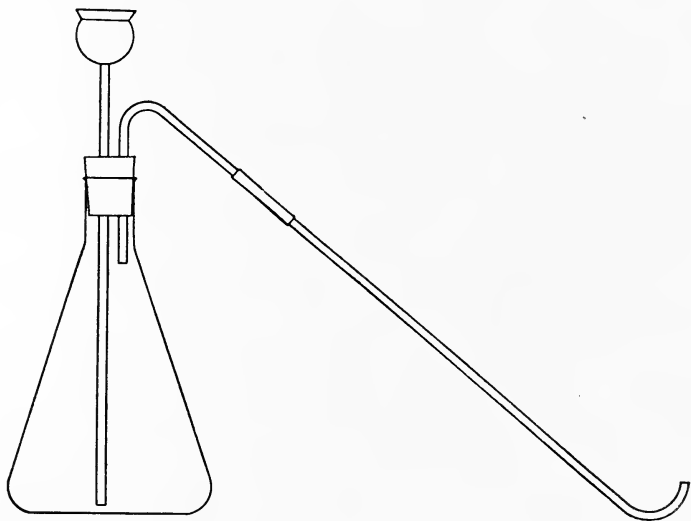


FIG. 7.

delivery of gas into bottles inverted in the pneumatic trough (Fig. 7). This apparatus must be air tight and may best be tested for tightness by closing the open end of the delivery tube with a rubber tube and pinch cock, then pouring water into the thistle tube. Explain why the water cannot run down into the flask if the apparatus is tight.

If the following directions are observed carefully there will

be no danger of an explosion, though as further protection a towel may be wrapped about the generating flask, if desired.

b. Place about 5 grams of granulated zinc in the generating flask, pour in enough dilute hydrochloric acid to cover the lower end of the thistle tube and collect the gas by holding an inverted test-tube over the end of the delivery tube. Carry the test-tube mouth downward to the Bunsen burner, *which must be at least two feet from the delivery tube*, repeating the process until the gas in the test-tube burns quietly. Try the effect if

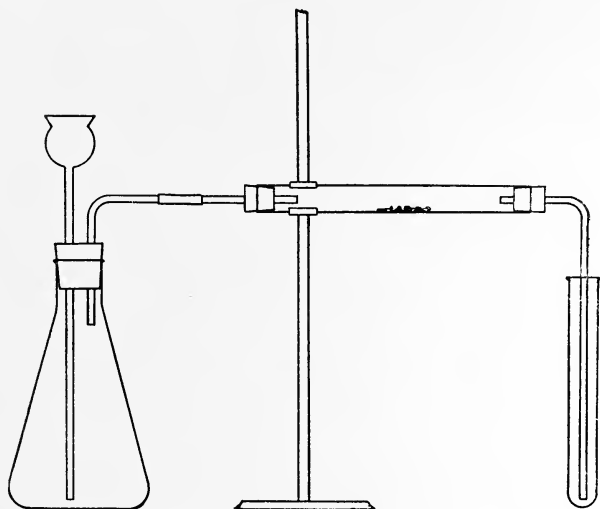


FIG. 8.

the test-tube of gas is carried mouth upward to the flame. What information does this give regarding the density of the gas? Collect a bottle of hydrogen by displacement of water and while holding it mouth downward insert into it a burning splinter. Why does the flame go out, and why is the splinter ignited again as it is withdrawn from the tube?

c. Attach a glass jet to the delivery tube by means of a short piece of rubber tubing. Collect a test-tube of hydrogen from this jet by displacement of air (should the test-tube be inverted or upright?) and after lighting the test-tube of gas at the Bunsen

flame carry it back quickly to the jet. If the hydrogen is pure enough to be lighted with safety it will light in this way. *Never apply a match to a hydrogen generator, but always light it with a test-tube of the burning gas as described.* Hold a watch glass above the flame for a moment. What is the deposit? Explain its formation.

d. Remove the glass jet and replace it with a hard glass tube open at each end and containing about 0.5 gram copper oxide (CuO), Fig. 8. After again testing the apparatus to make sure it is free from an explosive mixture warm the copper oxide gently while passing hydrogen over it. Hold a watch glass at the outer end of the combustion tube. What is deposited? What remains in the combustion tube? What has been reduced during this action? What has been oxidized? Write the reaction.

e. Clean out the hard glass tube and repeat *d* using magnetic oxide of iron in place of cupric oxide, and observing all precautions to avoid an explosion. Write the equation for this reaction. What has been oxidized during this action?

f. May other metals and acids be used in the preparation of hydrogen? Test this by pouring dilute sulfuric acid on samples of magnesium, aluminium and copper, each in a separate test-tube. The gas evolved may be tested by bringing the mouth of the test-tube to the flame. Add a few drops of dilute nitric acid to a sample of lead in a test-tube. Does the gas evolved have the properties of hydrogen? In all these cases if the reaction is not spontaneous the test-tube and its contents should be warmed gently before drawing any conclusions.

14. The Rate of Diffusion of Gases.

Draw out the closed end of a test-tube to a capillary (if the closed end gets too hot to hold, a piece of glass tubing may be sealed to it by heating the two together for a moment) cut the capillary, and reduce the size of the opening by heating, gently, until when the tube full of air is inserted open end down into a

beaker full of water (Fig. 9) it takes about one-half minute for the water to rise in the tube to a mark made about two inches from the open end. The water in the beaker must of course come above this mark on the test-tube. In order that the following measurements may be comparable the test-tube should always be brought to the bottom of the beaker, tipped just enough so there is free access of water from the outside, and the level of the water in the beaker should be always the same. Now determine accurately the time elapsing between the moment when the tube is inserted into the beaker of water and when the water comes to the mark on the test-tube, repeat this several times with the tube full of air, recording each determination, then fill the tube with hydrogen by displacement of air and again determine several times the time required for the water to rise to the mark, as before. Remember that in order to completely fill this tube with the easily diffusible hydrogen a rapid stream of the gas should be led into it, and in order to keep the hydrogen after the tube is filled the thumb may be placed over the open end, but the time elapsing between filling the tube and inserting it into the water must be as brief as possible. Repeat, filling the tube with carbon dioxide from a Kipp generator, observing all the precautions as in the case of hydrogen. Calculate the densities of these gases from their rates of diffusion, taking hydrogen as 1, and compare with the values given in the table.

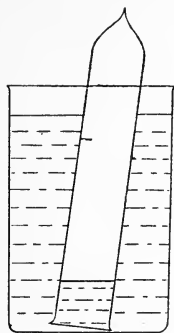


FIG. 9

15. The Combining Weight of Copper. Quantitative.

a. Fit up the hydrogen generator as described in Exp. 13, and connect the delivery tube with a hard glass combustion tube open at each end, held horizontally by the iron clamp, and from which a right angle bend leads to the bottom of a test-tube, Fig. 8. After proving the apparatus to be air tight disconnect

the combustion tube, place in it about 0.5 gram (do not weigh accurately) of powdered copper oxide (CuO), and again connect with the generator. Place about 7 grams of granulated zinc in the generator and add dilute hydrochloric acid till the lower end of the thistle tube is covered. *See that the delivery tube is at least two feet away from any flame.* Collect a test-tube of the gas by displacement of air, and carry it, inverted, to the flame, repeating the process until the gas in the test-tube burns quietly. Why? Now heat the copper oxide gently, having the delivery tube bent down and leading to the bottom of a test-tube. Notice the change in the copper oxide, and continue the heating until no further change takes place (this will require about 20 minutes), and all the water formed has been driven out of the combustion tube into the test-tube, then allow the tube and contents to cool while hydrogen is still passing through. (Be sure that the evolution of hydrogen continues through all the heating.) Write the equation for the reaction. What is the reducing agent in this action? What is the oxidizing agent?

b. If the copper oxide has been completely reduced, and the product resulting allowed to cool in an atmosphere of hydrogen, it should be pure copper. A weighed amount of this copper may now be converted to the oxide, weighed, and from these two weights the weight of oxygen in the oxide is obtained by difference. The combining weight of copper, or the weight of copper which will combine with 8 grams of oxygen, may be calculated by proportion.

In order to do this, clean and weigh a porcelain crucible. Place in it part of the copper obtained above, and weigh again. Now heat the crucible and contents until the red copper color has entirely disappeared, stirring occasionally with the platinum wire. Allow the crucible to cool, and weigh it. Heat it again and weigh again, until two successive weighings are within 2 mg. the same. Calculate the combining weight of copper.

16. The Equivalent Weight of Magnesium, Exp. 93, page 98, may be Performed here if Desired.

CHAPTER V

WATER AND HYDROGEN PEROXIDE

17. Composition of Water.

a. The qualitative composition of water by synthesis has been illustrated. Review the experiments.

b. The qualitative composition of water by analysis may be illustrated by the decomposition of water vapor by hot iron filings.

Fit a 100 c.c. flask with a stopper carrying a thistle tube and an obtuse angle delivery tube, which should be connected with a

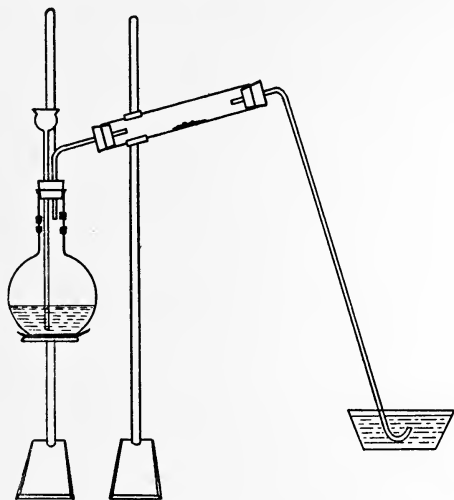


FIG. 10.

hard glass combustion tube, the further end of the combustion tube being connected with a delivery tube leading under water, Fig. 10. Make all connecting tubes as short as possible so as to avoid condensation of the steam. After making sure that the apparatus is air tight place about 50 c.c. of water in the flask and about a gram of iron filings in the combustion tube. Clamp the tube so that it slopes down toward the flask slightly (to prevent water from condensing and running over the hot

part of the glass) and heat the iron filings with one Bunsen burner, at the same time bringing the water in the flask just to boiling with another burner. For successful results the iron must be kept at a low red heat while steam is passing *slowly* over it. Collect the decomposition product of the steam by displacement of water, and test for hydrogen. What was the source of the hydrogen? Where will you expect to find the oxygen? Remove some of the material from the combustion tube. Does it appear the same as the iron filings used? Refer to the text-book for the composition of the iron oxide, and write the equation.

Compare this equation with the one written in Exp. 13 *e*. What is the relationship between these two equations? What kind of a reaction is this? What causes the equilibrium to shift in opposite directions in these two experiments?

18. Solubility of Salts in Water. Quantitative.

The solubility of a salt is defined as the weight of salt in 100 grams of a saturated solution at the given temperature. The solubility determination is usually made by weighing a given amount of solution known to be saturated at a given temperature, removing the water by spontaneous evaporation and finally by heating, then weighing the dry salt.

a. Cover each of two evaporating dishes with a watch glass or glass plate and weigh the dish with cover.

b. Prepare a saturated solution of potassium dichromate ($K_2Cr_2O_7$) or potassium chloride (KCl) by adding the powdered salt to about 50 c.c. of warm (40°) water until some remains undissolved after stirring intermittently for ten minutes. The success of this experiment depends on the preparation of a truly saturated solution, therefore plenty of time must be allowed for all of the salt that will to dissolve; the temperature must be kept as constant as possible, the solution must be stirred, and the salt must be powdered so that it will dissolve rapidly. Pour about 10 c.c. (estimate the quantity, do not measure it) of this

saturated solution into one of the weighed evaporating dishes, *noting the temperature of the solution at the moment of pouring*. Weigh the solution in the covered dish immediately, and only to the second decimal place, *i.e.*, record the weight as soon as you come to the place where 0.01 gram more is too much. Then place the dish in a safe place in the desk and remove the cover, leaving it to evaporate spontaneously, or hasten the evaporation by placing the dish on the steam bath. In the meantime the saturated solution will have cooled down, and, after stirring vigorously and allowing the crystals to settle, a second sample should be placed in the second weighed dish, the temperature *at the time of pouring* noted, and the proceeding repeated.

c. After the solutions in the dishes appear to have evaporated to dryness replace the covers and heat each one gently with the Bunsen burner, *keeping the cover on* to avoid loss from decrepitation of the crystals. Decomposition will result if potassium dichromate is heated above its melting-point. If moisture collects on the cover warm until it has entirely disappeared, then allow to cool and weigh.

d. From the data calculate the weight of potassium dichromate or potassium chloride which will dissolve in 100 grams of water at the two temperatures. Look up the solubility of the salt used at 10° and 20°, and draw a solubility curve from these four points.

19. Supersaturated Solutions.

a. Define a *solvent*, a *solute*, saturated, unsaturated and supersaturated solutions. Could a solution in contact with the solid solute remain permanently unsaturated or supersaturated? How would you make a supersaturated solution of a salt more soluble in hot water than in cold? Of a salt more soluble in cold water than in hot? Give two examples of unstable equilibrium and show in what respect each differs from stable equilibrium.

b. To 25 c.c. of water add 20 grams of hydrated sodium sulfate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and warm with stirring until solution is

complete. Decant into a perfectly clean beaker, cover with a watch glass and set aside, allowing the solution to cool spontaneously. Agitating the solution, or the presence of any solid matter, may cause crystallization to take place during the cooling. When the solution has cooled to room temperature, if no crystallization has taken place, drop in one crystal of sodium sulfate, and explain the phenomenon. If crystallization occurs during cooling the crystals will have to be redissolved by warming again, and the cooling and subsequent treatment with a crystal of sodium sulfate repeated. What is the condition of the solution (saturated, unsaturated or supersaturated) (1) when poured into the clean beaker, (2) when the undisturbed solution has cooled, (3) after crystallization has taken place? What test could be applied to determine whether a given solution is saturated, supersaturated or unsaturated?

20. Water of Hydration.

Heat 1 gram samples of copper sulfate, potassium nitrate, sodium chloride, sodium sulfate and potassium dichromate each in a separate test-tube, and determine which of them contain water of hydration. Place the samples of copper sulfate and sodium sulfate, after they have been heated in the test-tube, on watch glasses, and on two other watch glasses place samples of the same materials which have not been heated. Label these four samples and allow them to stand over night in the desk, then examine. In which case has the change brought about by heating been reversed? To what is this reversal due? Which of the salts is efflorescent and which deliquescent? Describe a means by which a deliquescent salt may be made to effloresce.

21. Natural Waters.

a. Evaporate about 5 c.c. each of distilled and tap water on watch glasses on the water bath, note the quantity of the residue in each case and test it to see if it is again soluble in water.

b. Test portions of tap and distilled water for chlorides by adding a few drops of dilute C.P. nitric acid (HNO_3) (side shelf)

and silver nitrate (AgNO_3) solution. (A white precipitate indicates the presence of chlorides.) Test other portions for carbonates by adding freshly filtered lime water (a white precipitate indicates the presence of carbonates) and for calcium salts by adding to fresh portions ammonium hydroxide (NH_4OH) and ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4$) solution (a white precipitate shows the presence of calcium salts). Compare the tests.

c. If rain water and sea water are available repeat these tests on them, and compare all four waters.

HYDROGEN PEROXIDE

22. Preparation and Properties of Hydrogen Peroxide.

a. To make a solution of hydrogen peroxide add gradually about 1 gram of sodium peroxide (Na_2O_2) (carry this in a watch glass or evaporating dish, as it will cause spontaneous combustion of organic material such as paper) to about 50 c.c. of water in a flask, which should be kept cool by holding under running water. Why? Then add dilute sulfuric acid, gradually and with stirring, until litmus paper placed in the solution just turns red, then test the solution as follows (save some of this solution for Exp. 23):

b. Prepare some lead sulfide by passing hydrogen sulfide through 1 c.c. of a solution of lead acetate ($\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$), or by adding a few drops of acetic acid to 1 c.c. of lead acetate, then adding 0.5 c.c. of ammonium sulfide. Add a few cubic centimeters of the hydrogen peroxide solution to this mixture, warm, and note the change in color of the precipitate. What is the reaction?

c. In another test-tube place 1 c.c. of a solution of potassium permanganate (KMnO_4), add a few drops of dilute sulfuric acid, and then the hydrogen peroxide. Test the escaping gas with a glowing splinter. What is it? Write the equation.

23. To Determine the Concentration of the Hydrogen Peroxide. Quantitative.

Fill a gas burette with water and attach a short-stemmed funnel to the rubber tubing above the stopcock (Fig. 11). Bring

the water up to the top of the graduated tube and close the cock, then introduce into the funnel 10 c.c. of the hydrogen peroxide solution prepared in Exp. 22, measured by the pipette. By opening the cock carefully the solution can be drawn into the gas burette without admitting any air after it. If air is admitted it must be forced out by raising the reservoir and opening the cock, but the mixing of the solution with the water in the burette which this causes is not desirable. Now place about 20 c.c. of potassium permanganate solution, which has been acidified with 2 c.c. of dilute sulfuric acid, in the funnel and draw about 2 c.c. at a time into the burette, until the color of the potassium permanganate in the burette is permanent,

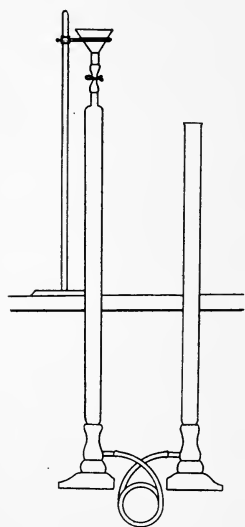


FIG. 11.

remembering that the solution can be drawn in only when the level of the water in the outer reservoir is below that in the burette, and *do not at any time draw in all the solution from the funnel*. When the reaction has ceased, as indicated by the permanence of the color, read the volume of oxygen liberated at atmospheric pressure, the barometer and the thermometer. Calculate the weight of oxygen obtained (remember that it was saturated with moisture). Write the equation for the reaction involved. What per cent of the oxygen liberated comes from the hydrogen peroxide? From what source does the remaining oxygen come?

Calculate the weight of the hydrogen peroxide which was present in the 10 c.c. portion of the solution. Assuming the solution to have a specific gravity of 1, calculate the per cent by weight of hydrogen peroxide in the solution.

24. Law of Multiple Proportion. Quantitative.

In experiment 8 (the law of definite proportion) the amount of potassium chloride in potassium chlorate was determined.

The amount of potassium chloride in potassium perchlorate may be determined in a similar fashion, and from these two determinations the amount of oxygen united with a given amount of potassium chloride in each of the compounds calculated. If the ignition tube and residue from Exp. 12 have been kept, the residue (the partially decomposed potassium perchlorate) should be heated until there is no further evolution of oxygen, then cooled and weighed. From the weights taken in Exp. 12. and this final weighing, calculate the per cent of potassium chloride in potassium perchlorate.

From the data obtained in Exp. 8 calculate the amount of oxygen which is combined with 1 gram KCl in KClO_3 . From the data in this experiment calculate the amount of oxygen which is combined with 1 gram KCl in KClO_4 . What is the ratio of these two amounts?

State the law of multiple proportion and show how this ratio illustrates it.

CHAPTER VI

THE HALOGEN FAMILY

25. Preparation of Chlorine.

Caution: *Do not allow chlorine to escape into the air unnecessarily, as the fumes are poisonous and cause very unpleasant colds if breathed in small quantities.*

By what general method may chlorine be prepared from hydrochloric acid? What sort of a reaction would this be? What general class of agents would be used for this purpose? What would you expect to be the result if materials which give oxygen on heating were warmed with hydrochloric acid? Test your conclusion by placing *very small* amounts of potassium chlorate (KClO_3), potassium perchlorate (KClO_4), and manganese dioxide (MnO_2) in separate test-tubes and warming with about 1 c.c. of dilute hydrochloric acid. The gas evolved should be tested for chlorine by holding a strip of paper wet with a starch potassium iodide solution (made by mixing equal volumes of starch solution and potassium iodide (KI) solution) above the mouth of the tube. If chlorine is present the paper will turn blue, due to the formation of a compound of starch and iodine which is blue, but a large amount of chlorine may further change the color to brown, then bleach it. The brown color is that of free iodine, and the bleaching is due to the oxidation of iodine by chlorine and water, forming iodic and hydrochloric acids. Write the reactions. As soon as the test has been made pour the contents of the tube down the sink, and follow with plenty of water.

26. The Density of Chlorine. Quantitative. *Poison.*

The method used in determining the weight of a liter of oxygen cannot be used in this case because of the solubility

of chlorine in water, and the lack of a material which can be used to give off chlorine as potassium perchlorate gives oxygen. The method used for chlorine in this experiment consists in weighing the same volume of air and of chlorine under known conditions, and finding the volume weighed by replacing the chlorine with a liquid, whose volume can be measured. The volume of the chlorine is then equal to the volume of the liquid necessary to replace it, and its weight is equal to the

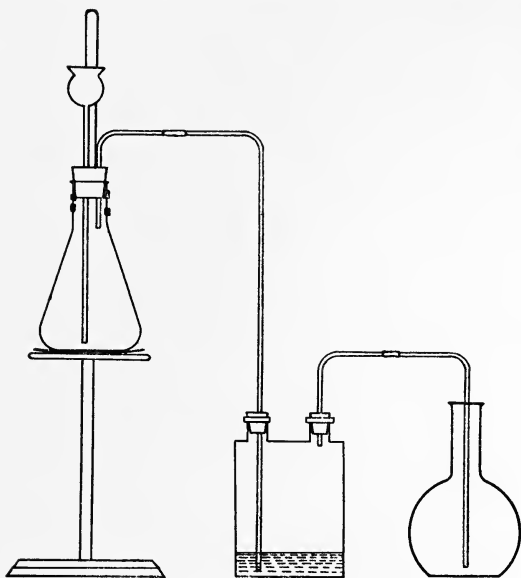


FIG. 12.

weight of that same volume of air + the difference between the weight of the flask and air, and the flask and chlorine.

a. Carefully clean and dry a 250 c.c. receiving flask, fit it with a cork stopper and weigh the flask and stopper, recording the atmospheric pressure and temperature at the same time. Now fit up a chlorine generator similar to that used for hydrogen, taking special care to have all rubber connections as short as possible, since chlorine attacks the rubber. The delivery tube from the generator should lead, through a cork, to the bottom

of a Woulff bottle, the other neck of which is fitted with a cork carrying a delivery tube which projects just below the surface of the cork, and is bent so that it will reach to the bottom of the receiving flask (Fig. 12). Place about 4 grams of manganese dioxide (MnO_2) in the generating flask and add 10 c.c. of concentrated hydrochloric acid, diluted with 10 c.c. of water. The chlorine coming from this generator should be dried by bubbling through concentrated sulphuric acid in the Woulff bottle (a depth of 1 cm. is sufficient) and should then be led to the bottom of the receiving flask to prevent diffusion into the air as far as possible. Generate the chlorine by warming gently and as soon as a green gas appears in the generating flask introduce the delivery tube into the weighed flask, leaving the flask unstoppered while chlorine passes in. As soon as the flask seems to be full of chlorine, as indicated by the color of the gas, remove the delivery tube and stopper the flask. Collect the rest of the chlorine in three gas-collecting bottles the mouths of which are closed as nearly as possible with glass plates, cover, and set aside for Exp. 27.

b. Weigh the stoppered flask containing the chlorine, again noting the atmospheric pressure and temperature, remove the cork and invert the flask over a beaker containing 225 c.c. of water and 25 c.c. of potassium hydroxide (KOH) solution. Do not allow the flask filled with chlorine to stand any length of time before this absorption, as the chlorine is absorbed by the cork, ruining the experiment. The chlorine will dissolve in the alkaline solution, causing the liquid to be sucked up into the flask. When the reaction seems to be complete place the beaker and flask, with its mouth still under the surface of the solution in the beaker, in a sink full of water, lower the beaker, bring the level of the liquid inside the flask even with the water outside, and while holding the flask in this position put in the stopper. Now remove the flask, and measure the volume of the solution it contains, which is equal to the volume of air replaced by chlorine.

c. The weight of the air replaced by chlorine may be calculated

from its volume, the temperature and pressure at the first reading (assume that the air was dry) and the weight of a liter of air under standard conditions. The difference between the weight of that volume of air and of chlorine may be found by subtracting the weight of the flask and air from the weight of the flask and chlorine, and that difference, plus the weight of the air, gives the weight of chlorine occupying the given volume under the second set of conditions. From this calculate the weight of a liter of chlorine.

27. Preparation and Properties of Chlorine. *Poison.*

a. If a supply of chlorine has not already been prepared in the previous experiment (26a.) it may be prepared by placing about 4 grams of manganese dioxide (MnO_2) in a generator similar to that used for hydrogen, adding through the thistle tube 10 c.c. of concentrated hydrochloric acid diluted with 10 c.c. of water, and warming gently. The delivery tube should reach to the bottom of a gas collecting bottle (should the bottle be upright or inverted?) which should be covered as completely as possible with a glass plate and the bottle should be replaced as soon as the greenish color of the chlorine becomes marked. Fill three bottles in this manner, cover and set aside and immediately place the delivery tube of the generator under a dilute potassium or sodium hydroxide solution; force the chlorine out of the generator and through the alkaline solution (what is the reaction between chlorine and the alkali?) by pouring water through the thistle tube until the generator is filled, then empty out the generator before proceeding.

b. Scatter a little powdered antimony into one of the bottles of chlorine. Write the reaction.

c. Place strips of moist and of dry colored calico in one bottle and account for the difference in behavior.

d. Drop moist pieces of red and blue litmus paper into the other, then introduce a jet of burning hydrogen (from a Kipp generator) until the appearance of the flame indicates that the chlorine is all used up, then again drop in pieces of

moist litmus. To what property of chlorine is the effect on litmus due? Why must the litmus be moist? What is the product when hydrogen burns in chlorine, and what is its effect on litmus? Would you expect to get the second test with litmus if any free chlorine was left in the gaseous mixture? Give the reason for your answer.

28. Hydrochloric Acid.

a. Review the experiment in which hydrochloric acid was prepared by synthesis.

b. Calculate the amounts of sodium chloride and sulfuric acid required by the equation $\text{NaCl} + \text{H}_2\text{SO}_4 = \text{NaHSO}_4 + \text{HCl}$ to give enough hydrochloric acid to fill two gas-collecting bottles, and to saturate 5 c.c. of water at 20° C. Place the calculated amount of sodium chloride in a generating flask fitted with a thistle tube and L-shaped delivery tube and add through the thistle tube a little more than the calculated amount of concentrated sulfuric acid (sp. gr. 1.84), diluted with one-half

its volume of water. In handling concentrated sulfuric acid be careful not to let it touch the skin, as it causes unpleasant burns; if any does get on the skin it should be washed off at once with plenty of water. In diluting sulfuric acid always pour the acid into the water. *Never pour water into concentrated sulfuric acid.*

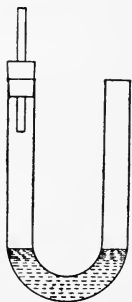


Fig. 13.

Place the generator on the wire gauze and warm gently, and with the delivery tube extending to the bottom of the gas collecting bottle, which should be dry, collect two bottles of the gas by displacement of air. The bottle is full of hydrochloric acid when moist blue litmus paper held just above the neck of the bottle turns red. Cover these bottles and set aside for future use. Then connect the delivery tube with a U tube (storeroom) as shown in Fig. 13. Place 5 c.c. of water in the U tube and continue the generation of hydrochloric acid

till the water is saturated. Save the generator and contents for future work.

c. Blow across the mouth of one of the bottles. Explain the formation of the fumes. Introduce into the same bottle a glass rod wet with ammonium hydroxide. What are the fumes in this case?

d. Invert the other bottle over water in the sink. What property of hydrochloric acid does this illustrate? If any gas is left in the bottle after shaking about with the water, test it to determine whether it is hydrochloric acid or air. Explain the test and results.

Why not collect the hydrochloric acid by displacement of water? How would you determine whether a solution of hydrochloric acid is saturated or not? What method should be used to determine the density of hydrochloric acid and why?

e. Place 1 c.c. of the solution from the U tube in a test-tube, and add a granule of zinc. What is the reaction? Pour the solution from the excess of the zinc and evaporate to dryness in an evaporating dish. What is the residue?

f. Add portions of the solution to a small amount of calcium oxide, until the latter is entirely dissolved. Evaporate this solution to dryness in an evaporating dish, and allow it to stand over night in the dish and examine. What is the reaction? What causes the change on standing?

g. Add a few drops of silver nitrate (AgNO_3) solution to another sample of the hydrochloric acid. What is formed? Where was this test used before? For what radical is it a test?

h. What are the products when hydrochloric acid acts on (a) metals, (b) oxides, (c) hydroxides, (d) oxidizing agents (Exp. 25)?

i. Pour the contents of the generating flask, *after cooling*, into a beaker containing about 10 c.c. of water (or if the contents of the flask solidify on cooling add 10 c.c. of water to the flask) and after allowing time enough for the solution to become

saturated (fifteen minutes at least) pour some of the solution from the solid residue and add a few cubic centimeters of concentrated hydrochloric acid. How does this experiment prove the reaction reversible? What means may be used to force the reversible reaction to completion?

29. Ionization.

a. Test the conductivity of each of the following by placing about 10 c.c. in a U tube, and inserting the platinum wires from the battery into each arm. If a current flows, the ammeter which is connected "in series" with the battery will indicate it by the position of the pointer. Rinse the platinum wires in distilled water between each test.

- (a) Distilled water.
- (b) Dilute hydrochloric acid.
- (c) Dilute potassium hydroxide (KOH) solution.
- (d) Dilute sodium chloride (NaCl) solution.
- (e) Alcohol and water.
- (f) Dilute sugar solution.

Notice the phenomena which take place at the poles when a current flows. Test to determine which pole gives hydrogen and which oxygen or chlorine. Which pole is the anode? What happens to the sodium ion when it loses its electrical charge at the cathode, when electrolyzing sodium chloride solution? Under what condition could metallic sodium be prepared by electrolysis of sodium chloride? The first products of the electrolysis, in this case sodium and chlorine, are called the primary products; any products resulting from a reaction of the primary products with other materials present, in this case, products from the action of sodium on water are called secondary products. Write equations showing the primary and the secondary products of each electrolysis.

b. To what classes of chemical compounds do conductors of the second class (those which conduct in solution, and at the same time undergo a chemical change) belong? How does the ionic theory explain the passage of the current through the

solution? What is the difference between the chloride ion and free chlorine as regards (a) color, (b) conduct when subjected to the influence of a charged plate or pole, (c) chemical properties. What sort of reaction is especially characteristic of those compounds which ionize in solution?

30. The Purification of Rock Salt, (NaCl).

Rock salt, as mined, is of course mixed with more or less of those salts which are in solution with it in sea water, for example sulfates and chlorides of calcium, magnesium, potassium, etc. The presence of sulfates in the crude salt may be shown by adding a few drops of barium chloride (BaCl_2) solution to a dilute solution of the salt acidified with C. P. hydrochloric acid. A white precipitate indicates sulfates. How may the presence of calcium salts be shown? (Exp. 21.)

a. Saturate 50 c.c. of water with common salt. From the table of solubilities the amount of salt required may be determined, and a little more than the theoretical amount should be taken to allow for moisture and foreign matter. Filter the solution and test small portions of it for sulfates and calcium. Divide the solution remaining into two equal parts, place one part in an evaporating dish and evaporate on a steam bath until about two-thirds of the water has evaporated, allow the salt to crystallize by cooling, then filter out the crystals, drain as completely as possible, wash them with a very small amount of distilled water, drain, label No. 1, and set away to dry. Into the other half of the solution pass hydrochloric acid gas, generated by the action of the calculated amount of concentrated sulfuric acid (sp. gr. 1.84) diluted with half its volume of water, on 10 grams of sodium chloride. The generating flask should be fitted with a stopper carrying a thistle tube reaching to the bottom of the flask, and a delivery tube dipping into the solution of sodium chloride to be purified. Heat the generating flask gently, keeping the stream of gas passing into the salt solution sufficiently rapid so that there is no tendency for the solution to suck back into the generator, and

continue until there is no further precipitation in the salt solution. Filter out the precipitate from the solution, drain, wash it with a very small amount of distilled water, and drain, finally allowing it to dry completely by evaporation. Label this sample No. 2. What is this precipitate? Now dissolve small portions of each sample in distilled water and test as before for sulfates and calcium. Judge the relative amounts of impurities in the three samples tested, and draw conclusions as to the efficiency of the two methods of purification. Test solutions of samples 1 and 2 with red and blue litmus paper. What has become of the hydrochloric acid added to sample 2? Is the precipitation of chlorides by hydrochloric acid a general property? Test your conclusion by adding 1 c.c. of concentrated hydrochloric acid to 1 c.c. of saturated solutions (make these by adding an excess of the solid to 1 c.c. of water, and after shaking five minutes pour off the clear solution from the excess of the solid) of barium chloride (BaCl_2) and potassium chloride (KCl).

31. Potassium Hypochlorite.

a. Dissolve 5 grams of solid potassium hydroxide (KOH) in 15 c.c. of distilled water. Cool the solution and place about half of it in a test-tube, reserving the remainder for Exp. 32. Fit up a small generating flask with a delivery tube and a thistle tube which reaches nearly to the bottom of the flask. Place about 4 grams of finely pulverized manganese dioxide (MnO_2) in the flask and add 10 c.c. of concentrated hydrochloric acid diluted with 10 c.c. of water, through the thistle tube. Warm the flask gently, and when the generation of chlorine begins introduce the delivery tube into the test-tube containing the potassium hydroxide solution. Surround the test-tube with cold water and stir the solution while chlorine is being passed into it. Keep a lively stream of chlorine flowing into the solution for ten to fifteen minutes, then stop the generation of chlorine by allowing the generator to cool down, remove the test-tube and immerse the end of the delivery tube in water or a dilute alkali solution. *Do not allow chlorine to escape into the*

air. This generator may be used again in Exp. 32, but it should not be allowed to stand over night in the desk.

b. Pour the contents of the test-tube into an evaporating dish and wet a piece of colored cloth in it. Is the cloth bleached? Place the cloth in a beaker containing some dilute sulfuric acid. If no change is observed wet the same piece of cloth with the two solutions again. Explain what happens. Why is sulfuric acid necessary? Explain why this treatment will bleach cloth while air does not. Write equations for the reaction in *a*, and for the action of sulfuric acid. Why was the test-tube cooled in *a*? Acidify a small portion of the solution from *a* with nitric acid, then add a few drops of silver nitrate solution. What ion does this show to be present?

32. Potassium Chlorate.

Using the same generator as in the preceding experiment (31) pass chlorine into the remainder of the potassium hydroxide solution but do not cool the tube. Continue this process until the solution is completely saturated with chlorine, then dispose of the generator and contents as in Exp. 27a. How is it possible to tell when the point of saturation is reached? Allow the mixture to cool. What happens? Filter and test the filtrate by adding nitric acid and silver nitrate. What substance is present? Dry the crystals and heat in a dry tube, testing for any gas that might be evolved. What are the crystals? Which is most soluble, potassium chloride or potassium chlorate? Confirm your conclusion by reference to the table. Write all reactions. Explain why this experiment and the preceding do not yield the same products.

33. The Preparation of Potassium Perchlorate.

When potassium chlorate is heated two reactions take place simultaneously: $4 \text{KClO}_3 = 3\text{KClO}_4 + \text{KCl}$ and $2\text{KClO}_3 = 2\text{KCl} + 3\text{O}_2$. The greater part of the potassium chlorate will go through the change represented by the first equation if the temperature is held just above the melting-point of the potassium chlorate, so that the evolution of oxygen (represented by the second

equation) is very slow. If the temperature becomes too high the evolution of oxygen is rapid, the amount of perchlorate formed is not so great, and it may itself decompose according to the equation, $\text{KClO}_4 = \text{KCl} + 2\text{O}_2$. Thus, in preparing potassium perchlorate from potassium chlorate the potassium chlorate must be heated just enough to keep it melted, and evolving a *slow* stream of oxygen.

Place about 8 grams of potassium chlorate (free from any combustible matter, why?) in the hard glass test-tube, and stopper with a cork or rubber stopper carrying a delivery tube leading to the water trough. After testing the apparatus for air tightness melt the potassium chlorate and heat it gently, collecting the oxygen evolved by displacement of water. Take care that the melted salt does not come in contact with the stopper. Why? Regulate the heat so that the evolution of oxygen is very slow, and the salt is kept melted throughout. When 500 c.c. of oxygen have been evolved withdraw the delivery tube and allow the melted mass to cool. From the volume of oxygen evolved calculate the weight of potassium chlorate which has decomposed according to the second equation. Calculate the amount of potassium chloride and potassium perchlorate which should now be in the reaction mixture, assuming that all the potassium chlorate has decomposed. Look up the solubilities of the two salts left and devise a method for separating these. After submitting your method for the approval of an instructor carry out the separation, wash the potassium perchlorate and weigh roughly. What per cent of the theoretical yield is obtained? Should the method of separation used give a pure perchlorate? Test a small portion for chlorides and save the rest of the product for Exp. 34.

34. To Test the Purity of the Potassium Perchlorate. Quantitative.

Place from 0.2 to 0.5 gram of the *perfectly dry* potassium perchlorate obtained in Exp. 33 in a weighed hard glass ignition tube, weigh accurately, then heat until the evolution of oxygen

is complete, and weigh the tube and residue. Calculate the per cent of oxygen in the given sample. Compare this result with that obtained in Exp. 24 and with the theoretical composition of KClO_4 . Was the potassium perchlorate pure?

35. The Preparation of Bromine.

Caution: Do not breathe the vapor of bromine. Do not get liquid bromine on the skin, as it causes severe burns.

Calculate the quantities of manganese dioxide (MnO_2), potassium bromide (KBr) and sulfuric acid necessary to make 2 c.c. of liquid bromine (sp. gr. 3.18). Have this calcula-

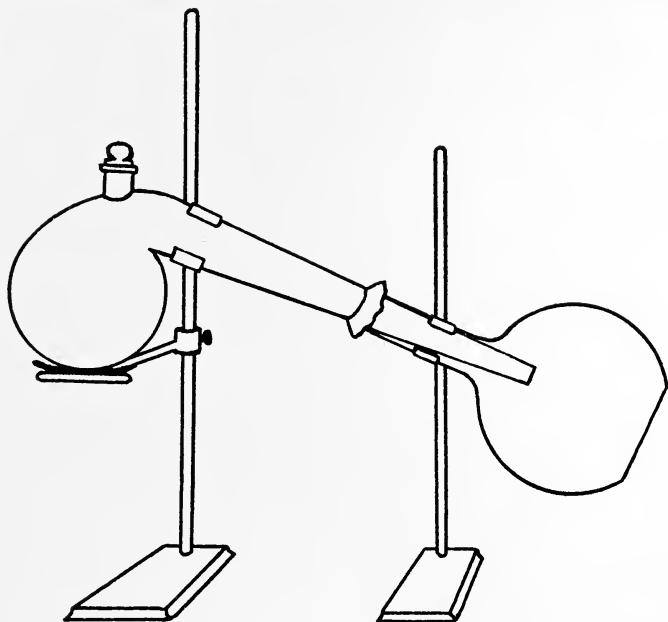


FIG. 14.

tion approved by an instructor before proceeding. The volume of concentrated sulfuric acid required (sp. gr. 1.84) should be diluted with one-half its volume of water by pouring the measured amount of concentrated acid into the measured amount of water in a beaker. *Never pour water into sulfuric*

acid. Grind up the required amount of potassium bromide in the mortar and mix with the manganese dioxide, then place the mixture in a retort (storeroom) and add the diluted sulfuric acid through the tubule. Close the tubule and heat gently, protecting the bottom of the retort by a wire gauze. Collect the bromine in a flask into which the neck of the retort extends as far as possible, and which is kept cool by being placed under a stream of running water, while the escape of bromine vapor into the air is prevented by folding a piece of wet filter paper over the mouth of the receiving flask (Fig. 14). When the evolution of bromine has ceased remove the flask and test the gas in the flask with pieces of paper wet with starch solution, and with starch potassium iodide solution (see Exp. 25). (Then proceed at once to Exp. 36. Do not allow the bromine to stand over night.) To what is the difference in color in the two tests due? Refer to the starch potassium iodide test for chlorine. Would you expect to find the same succession of colors in this case? Write equations for the reactions involved. Could bromine be prepared by any of the methods used for chlorine? If potassium iodide was present as an impurity in potassium bromide how could the potassium bromide be most readily purified? Is there any connection between the low boiling-point of bromine and the fact that the air above the liquid bromine is strongly colored with bromine vapor at room temperature?

36. Potassium Bromate.

a. Cover the bromine collected in the preceding experiment with 2 to 4 c.c. of water and add drop by drop a strong solution of potassium hydroxide (KOH, 1:1) with shaking, until the liquid bromine has entirely dissolved, and the color of the solution is only faintly yellow. What reaction is taking place? (Note: If a brown precipitate of manganese dioxide, carried over mechanically by the bromine, appears in the solution on the addition of potassium hydroxide it must be filtered out before the color of the bromine in solution is entirely removed by the

potassium hydroxide.) Heat this solution to boiling, (what further reaction occurs?), and continue boiling until crystals begin to separate out, then cool under running water, and filter after the solution has come to room temperature. Rinse out the flask and the precipitate on the filter paper with 2 c.c. water and save the filtrate. Allow the crystals to drain as completely as possible and then in order to purify them wash into an evaporating dish with a fine stream of distilled water from the wash bottle. Heat till the crystals are entirely dissolved, then allow the solution to crystallize spontaneously by cooling and evaporation. Remove the crystals from the solution before all the water has evaporated and dry them on filter paper, then place in a weighed (quantitative) test-tube and label in such a way that the label can be readily removed before a second weighing. Boil the filtrate from the first crystallization (in an evaporating dish) until it begins to crystallize, then allow to cool. Remove the crystals, dry them on filter paper, and place them in another weighed test-tube, labeling as before. These may be best labeled according to solubility. Which crystals represent the least soluble, and which the most soluble portion? Write the reaction involved when bromine acts on potassium hydroxide under the given conditions, and after looking up the solubilities of the products determine which salt you will expect to find in the least, and which in the most soluble portion of your product. Weigh the test-tubes with the salt, and by subtracting the weight of the empty tubes get the weights of the salts.

b. Devise some method of testing these two salts to determine whether your conclusions regarding their identity are correct. *Get the approval of an instructor* before proceeding to apply the tests you devise. Compare the reaction between bromine and potassium hydroxide with that used in Exp. 32 between chlorine and potassium hydroxide. Calculate from the original amount of KBr taken in Exp. 35, the theoretical yields of KBrO_3 and KBr. What per cent of the theoretical yield is

the amount obtained; or in other words, what is your percentage yield? At what stages of the process has there been an opportunity for loss of material?

37. Iodine.

Mix together a small crystal of potassium iodide, and a little manganese dioxide. Place the mixture in a test-tube and add a few drops of concentrated sulfuric acid, then heat gently. Note the color of the gas evolved. Is it lighter or heavier than air? What observation leads to this conclusion? Hold a paper wet with starch solution in the gas. Explain. Why was potassium iodide used with the starch solution in the test for chlorine and bromine and not in this test? Compare this method for the preparation of iodine with those for chlorine and bromine. Why would it not be safe to use potassium chlorate as the oxidizing agent on a mixture of potassium iodide and concentrated sulfuric acid (see test-book under chlorine dioxide). What would be the reaction between KClO_3 and HI ?

38. Hydrofluoric Acid and its Etching Action on Glass.

Prepare hydrofluoric acid by placing 2 grams of calcium fluoride (CaF_2) in a lead dish, or evaporating dish which has been coated with paraffin, and mixing with enough concentrated sulfuric acid to make a paste. Cover the dish with a glass plate which has been protected by a coating of paraffin except in the places which are to be etched. Allow the dish and cover to stand for about an hour, then remove the paraffin from the cover glass and examine. Write the reactions and account for the disappearance of part of the glass. Test the gas in the dish by breathing across it. What are the fumes? Hold a rod wet with ammonium hydroxide (NH_4OH) above the dish. What are the fumes? Is this a distinctive test for hydrofluoric acid? For what other acid has it been used? What one property is essential that an acid should give this test?

39. Equilibrium.

a. Review the preparation of hydrochloric acid and show how the reaction was proved to be reversible. What property of the hydrochloric acid made it possible to get complete conversion of the sodium chloride into hydrochloric acid, even though the reaction was reversible?

The reaction, $\text{NaH}_2\text{PO}_4 + \text{HCl} = \text{NaCl} + \text{H}_3\text{PO}_4$, is reversible, and if at any fixed temperature solutions of primary sodium phosphate and hydrochloric acid, or of sodium chloride and phosphoric acid of equivalent concentrations are mixed, the reaction mixture will consist of a solution of the two original materials and the two resulting products in exactly the same proportions, regardless of which pair of materials was originally used.

b. To show this experimentally make an indicator by mixing six volumes of potassium iodide solution with one volume of potassium bromate solution, and add a few drops of this indicator to small portions of normal hydrochloric and phosphoric acid solutions, noting the effect of the indicator on the two acids. Now prepare four tubes, in one placing equal quantities of normal solutions of primary sodium phosphate and hydrochloric acid, in another the same quantities of normal solutions of sodium chloride and phosphoric acid, in the third the same quantities of normal hydrochloric acid and water, in the fourth the same quantities of normal phosphoric acid and water, and label each tube. Shake each thoroughly then add an equal number of drops¹ of the indicator to each (the two diluted acid solutions are to be used as standards to show the color given by hydrochloric and phosphoric acids of the same concentration as in the first two tubes). From the color reactions (these must be noted at once, as the color deepens on standing) draw conclusions as to the composition of the solution in each of the first two tubes.

¹Each student should provide himself with half a dozen pieces of glass tubing, drawn to a jet at one end and kept clean and dry to use as dropping tubes.

c. Add twice normal phosphoric acid to an equal volume of normal sodium chloride and note the color given by the same number of drops of the indicator as before. Compare with the tube made from normal phosphoric acid and sodium chloride. What effect does increased concentration of one of the reacting substances have on the point of equilibrium?

CHAPTER VII

SULFUR

40. Physical Properties of Sulfur.

What is a polymorphous substance? Do the polymorphic forms of a given substance differ most with respect to physical or chemical properties? Illustrate. Place about 5 grams of sulfur (sample A) in a short, wide, dry test-tube and heat gently till it just melts (use special care not to heat the sulfur much above its melting-point), then pour a small portion of the melted sulfur into a beaker containing cold water (sample B). Continue the heating of the rest of the sulfur in the test-tube until it begins to boil, noting the changes in appearance and viscosity as the temperature rises. Pour the boiling sulfur into another beaker of cold water (sample C). Compare these three samples of sulfur (A, B, C) as to appearance, texture and solubility in carbon disulfide. To study this last property place a small portion of each sample in separate test-tubes and add 1-2 c.c. of carbon disulfide (CS_2), then shake, taking care that the test-tubes are at a safe distance from all flame, as carbon disulfide is very inflammable, and its vapor mixed with air is explosive. Allow small portions of these solutions to evaporate spontaneously. Did any sulfur dissolve? Record the results; then pour the contents of the test-tubes down the sink, following with plenty of water. Let portions of samples B and C stand in the desk over night and examine again in the same way. Explain the differences in the behavior of the three samples. Retain the test-tube in which the sulfur was melted for use in the next experiment (41 b.)

41. Chemical Properties of Sulfur and Hydrogen Sulfide.

a. Mix 1 gram powdered zinc and 0.5 gram sulfur, place on a crucible cover and heat. *Caution.* What is the reaction?

b. Mix 2 grams of iron filings and 1 gram of sulfur, place in the test-tube used in Exp. 40 and heat. What is the reaction?

c. What reactions of chlorine and oxygen are analogous to these for sulfur? From the position of sulfur in the periodic table would you expect it to be like these two elements? Examine these two products by placing samples of each (obtain the iron compound by breaking the tube) in separate test-tubes and adding a few c.c. of dilute hydrochloric acid. Note the odor of the gas evolved (cautiously). Test it with paper moistened with lead acetate solution. Write the reactions involved.

d. Introduce a small portion of burning sulfur on the iron spoon into a gas-collecting bottle, and cover the bottle as completely as possible with a glass plate. What is the reaction? When the reaction has ceased place 10 c.c. of water in the bottle and shake. What is formed? Pour this solution into a test-tube and pass in hydrogen sulfide, which may be generated by placing some of the iron or zinc sulfide in a test-tube, adding dilute hydrochloric acid and closing the tube with a stopper carrying a delivery tube which can be inserted into the solution. Note the appearance and odor of the solution after the reaction has taken place. Write the equation.

e. This is an example of a colloidal solution, and is called "milk of sulfur." Two of the general properties of colloidal solutions may be easily illustrated by pouring the solution through a filter paper, showing the fine state of division of the sulfur, then adding a few crystals of sodium chloride, shaking and filtering again. The electrolyte has coagulated the colloid.

f. Pass some hydrogen sulfide, generated in the same way, into 10 c.c. of water, stopper and allow the solution to stand over night. What is formed? What is the oxidizing agent in this case? These reactions have been suggested to explain the occurrence of free sulfur in volcanic regions.

g. Pass hydrogen sulfide from a Kipp generator or from the

laboratory supply into a solution of a zinc salt, for example zinc chloride. Is the reaction reversible?

42. Sulfur Dioxide.

a. If fresh solutions of ferric chloride (FeCl_3) and potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) are mixed, and the brown solution so obtained is taken up on filter paper, it may be used as an indicator for sulfur dioxide (SO_2), as the color changes to a bluish-green when exposed to the gas. The color changes in the same way if the paper is exposed to air and light for any length of time, so it must be prepared and used for the test at once. If this is done there is no difficulty in recognizing appreciable amounts of sulfur dioxide.

b. Heat 2 c.c. of concentrated sulfuric acid in contact with copper turnings in a test-tube until there is a spontaneous evolution of gas. Test the escaping gas with the freshly prepared paper. What is formed? Write the reaction. Does dilute sulfuric acid act on copper? (See Exp. 13.) Allow the tube and contents to cool, then pour the contents in a beaker containing about 50 c.c. of water. Allow the precipitate to settle, and explain the color of the solution.

c. Add concentrated sulfuric acid to a small crystal of potassium bromide (KBr) and heat gently, testing the escaping gas as before. Explain the color of the evolved gas in this case. Write the reactions taking place.

d. What name would be given to any process by which SO_2 is obtained from H_2SO_4 ? In these two experiments what are the reducing agents? What is the oxidizing agent?

e. Add some dilute hydrochloric acid to a few sodium sulfite (Na_2SO_3) or sodium acid sulfite (NaHSO_3) crystals in a test-tube, warm and test for sulfur dioxide. Write the reaction. Fit this tube with a cork and delivery tube, and pass the gas evolved (use care that none of the liquid is carried over) into a test-tube containing 9 c.c. of water and 1 c.c. ammonium hydroxide (NH_4OH) until the solution becomes saturated. Now pass the gas into another test-tube containing 1 c.c.

potassium permanganate (KMnO_4) solution and 9 c.c. water. Note the change in appearance. (What other reagent has the same effect on KMnO_4 ?)

Compare the two solutions by adding to each a little barium chloride (BaCl_2) solution, shake and note the effect, then allow to settle, decant the clear liquid and add dilute C.P. hydrochloric acid to the precipitate in each tube. How do you account for the difference in behavior? Write equations for all the reactions involved.

43. Preparation and Properties of Sodium Thiosulfate.

Dissolve 10 grams of anhydrous sodium carbonate in 50 c.c. of water, warming if necessary, or if only the hydrated salt ($\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$) is available dissolve a quantity of it equivalent to 10 grams of Na_2CO_3 in a correspondingly smaller amount of water. Divide this solution into two equal parts, and saturate one part with sulfur dioxide, generated by heating 10 grams of copper turnings with 20 c.c. concentrated sulfuric acid in a generating flask provided with a thistle tube and delivery tube. *Be particularly careful to see that the solution of the carbonate is not at any time sucked back into the generating flask.* Add to the solution prepared in this way (what is it?) the other part of the sodium carbonate solution, and heat to boiling. What two reactions have occurred thus far? Write equations for each. After the effervescence has ceased (to what is it due?) add 4 grams of sulfur, boil for 20 minutes keeping the volume of the solution constant, filter off the excess of sulfur, evaporate the solution over a water bath until crystallization commences, then allow it to cool. Filter off the crystals and evaporate the filtrate to crystallization a second time, then cool and combine the two crops of crystals. Weigh the product (after drying) on the rough laboratory balance, and calculate the percentage yield. Dissolve some of the crystals in water, and test the solution with blue and red litmus paper. Hydrolysis of the salt into a base and acid has taken place. According to the test, which (base or acid) is more highly

ionized? Add a few drops of hydrochloric acid to the solution and warm. The precipitate is milk of sulfur, where have you seen this before? Write the equation for the reaction. Is thiosulfuric acid a stable compound? Add a solution of sodium thiosulfate to some freshly prepared silver chloride and to a small crystal of iodine. Write the reactions. Of what technical use is sodium thiosulfate?

44. Sulfuric Acid.

a. Study the properties of sulfuric acid by the following tests: Dip a strip of filter paper into dilute sulfuric acid and dry on the radiator, then examine. Add a few drops of concentrated sulfuric acid to a little sugar in an evaporating dish. Compare with the effect of heating sugar on a crucible lid. Add 1 c.c. of concentrated sulfuric acid to 1 c.c. of water in a test-tube, noting the temperature before and after mixing. Does the last experiment throw any light upon the nature of the change in the first two? Explain.

b. Add barium chloride (BaCl_2) solution to dilute sulfuric acid. What is the reaction? Allow the precipitate to settle, pour off the clear liquid and add a few cubic centimeters of concentrated C. P. hydrochloric acid to the precipitate. Is the above reaction appreciably reversible under these conditions?

45. Neutralization. Determination of the Concentration of a Solution of Acid or Base. Quantitative.

The neutralization of an acid by a base depends on the union of the hydrogen ions of the acid with the hydroxyl ions of the base to form unionized water. If an indicator, litmus, is present in a solution of an acid it will give the solution a red color, due to the H^+ ions. If a base, as KOH , is added to that acid solution, the H^+ ions are gradually removed by the OH^- ions of the base, until finally there are none left in the solution, and the addition of the next drop of the solution of the base causes the litmus to turn blue, because of the free OH^- ions in the solution. Such a solution, the addition of one drop of

base or acid to which causes it to turn litmus blue or red, is said to be neutral, and is free from both H^+ and OH^- ions except to the extent to which they occur in water, where they are equal in number. What ions are present in a solution of H_2SO_4 which has been neutralized by KOH ? Write the ionic equation for the reaction.

The concentration of an acid solution may be determined by neutralizing a measured volume of the solution, evaporating to dryness and weighing the salt formed (it must be a non-volatile, stable salt, why?) and calculating from the weight of the salt, the weight of the acid originally in the solution.

a. Make a dilute solution of sulfuric acid by pouring 3 c.c. of concentrated sulfuric acid into 100 c.c. of water and mix thoroughly by shaking. Place in a flask and stopper the flask, so that the solution will not change in concentration by evaporation. Dissolve about 6 grams of solid potassium hydroxide (KOH) (use care in handling this, as it is a caustic and burns the skin) in 100 c.c. of water, shake thoroughly and stopper. Label the flasks.

Save these solutions as they must be used again in Exp. 46.

b. Rinse out and fill a burette (storeroom) with the prepared potassium hydroxide solution drawing enough solution out through the nozzle to insure the removal of all the air from the fine tubing, and bring the solution in the burette to the zero mark. Now make a rough determination of the amount of potassium hydroxide solution required to neutralize 10 c.c. of the sulfuric acid solution as follows: Measure out 10 c.c. of the sulfuric acid solution with the pipette, and place it in a beaker, add 2 drops of phenolphthalein (which is an indicator like litmus, except that it is colorless in acid and red in alkaline solutions) and then while stirring continuously, add the potassium hydroxide solution from the burette, 1 c.c. at a time, until the solution in the beaker becomes permanently red, *i.e.*, until it is alkaline. Read the burette. The volume of potassium hydroxide solution used is not more than 1 c.c. greater than the volume required for exact neutralization of

10 c.c. of the sulfuric acid solution if these directions have been carefully followed. Now make the exact, final determination as follows:

c. Clean and weigh (quantitative) an evaporating dish and cover, then place in it exactly 10 c.c. of the sulfuric acid solution, measured with the pipette, and weigh again. From the weight of the solution and its volume calculate its specific gravity. Add a couple of drops of phenolphthalein and after reading the burette neutralize the acid with potassium hydroxide from the burette, adding at once 1 c.c. less than the amount required in the rough neutralization, then to determine the fraction of a cubic centimeter required to just neutralize the acid add the potassium hydroxide drop by drop, stirring constantly with a glass rod, and stop the addition the moment the solution turns permanently red. The color is best seen against a white background.

When the solution is just neutral read the burette again, and determine the volume of potassium hydroxide solution used. Evaporate the neutral solution to dryness on a steam bath, or by setting it above a beaker of boiling water, and when dry heat the covered dish gently with the direct flame, cool and weigh. Heat the dish and contents again, and weigh, until two successive weighings give the same result. What is left in the dish?

d. Define per cent composition and normal solution. From the weight of the salt formed calculate the weights of sulfuric acid and potassium hydroxide used. Calculate the normal strength of the sulfuric acid and potassium hydroxide solutions. How many milligrams of sodium hydroxide (NaOH) will 1 c.c. of the sulfuric acid neutralize? How many milligrams of ammonium hydroxide (NH_4OH)? How many milligrams of nitric acid (HNO_3) will 1 c.c. of the potassium hydroxide neutralize? Of hydrochloric acid? Of sulfurous acid (H_2SO_3)? Calculate the per cent composition of the sulfuric acid solution. What further information would be necessary for you to calculate the per cent composition of the

potassium hydroxide solution? Why could not the concentration of these solutions be determined by evaporating off the water and weighing the residue, as in Exp. 18?

46. Potassium Acid Sulfate.

Place 10 c.c. of the dilute sulfuric acid prepared in Exp. 45 (measured with the pipette) in a clean evaporating dish, then add, from the burette, just half as much potassium hydroxide solution as was found necessary for neutralization, and evaporate the solution to dryness on the steam bath. What is formed? Dissolve a few of the crystals in water and test the solution with red and blue litmus paper. Explain the effect. Dip a filter paper into the solution, and allow it to dry out on the radiator. Is sulfuric acid present in the solution? If sulfuric acid is not found, how can the effect on litmus be explained? Add some of the solution to some solid sodium carbonate in a test-tube. Explain. Add barium chloride (BaCl_2) to the solution, then hydrochloric acid. Is the sulfate ion present?

CHAPTER VIII

NITROGEN

47. Preparation and Properties of Nitrogen and Ammonia.

a. What are the principal constituents of the air? Are they present as a mixture or in chemical combination? How may oxygen be removed from the air?

b. Float a crucible on the trough full of water, place in it about 0.5 gram red phosphorus and light it by touching it with the hot end of a file or iron spoon. Quickly invert a gas-collecting bottle over the dish, bringing the mouth of the bottle below the surface of the water, and hold in that position till the phosphorus has ceased burning, and the water inside the bottle has come to a fixed level. Account for the rise of the water in the bottle. Place a glass cover over the mouth of the bottle while it is still under water, and withdraw. Test the gas in the bottle by inserting into it a burning splinter. Does it support combustion? Does it burn? Insert into it a piece of burning magnesium ribbon. Does it support combustion in this case? What is formed?

c. Place about 0.5 gram of magnesium powder in a crucible, cover and heat while covered until all glowing has ceased. The crucible contains a mixture of what two compounds? Place the contents of the crucible in a test-tube, moisten with water, and warm gently. Test the escaping gas with moistened red litmus paper. What is it? Write the reactions.

d. To 1 c.c. of ammonium chloride (NH_4Cl) solution in a test-tube add a few drops of potassium or sodium hydroxide solution, and warm, testing the gas evolved as before. What is it? Write the reaction. Hold a rod wet with concentrated hydrochloric acid at the mouth of the test-tube. Explain the fumes formed. Devise and apply a test to determine whether the gas is lighter or heavier than air.

48. The Weight of a Liter of Ammonia. Quantitative.

Generate ammonia by warming together in flask fitted with a delivery tube, and proved to be air tight, a mixture of equal weights of ammonium chloride and powdered quick lime (CaO). Collect three test-tubes of the gas by air displacement and test its solubility in water, dilute alkalis (dilute sodium hydroxide or potassium hydroxide) and dilute acids. From this test determine which will make the most suitable liquid for absorbing the gas, then proceed with the determination of the weight of a liter, according to the method used for chlorine. Could concentrated sulfuric acid be used to dry this gas? Why? If the gas is prepared as directed, from dry ammonium chloride and quicklime it will not be necessary to dry it, but it may be passed directly into the weighed flask. Should the gas be collected with the flask held upright or inverted? The flask is sufficiently filled when a strip of moistened red litmus paper held at the mouth turns blue.

49. Ammonium Hydroxide.

a. Attach a U tube containing water to the ammonia generator used in the previous experiment, similar to the arrangement used for making a solution of hydrochloric acid (Fig. 13) and pass in ammonia until the solution is saturated. Note the effect on the temperature of the solution. Test this solution with litmus, then boil a small portion in a beaker for 5 minutes and test it again. Explain. Neutralize another portion of the solution with dilute hydrochloric acid and evaporate the neutral solution to dryness on the water bath, then heat the dry residue with the direct flame. Repeat this procedure only neutralize with dilute sulfuric acid instead of hydrochloric. Account for the difference in behavior on heating the dry salts. Is there any relation between the volatility of the acid and of its ammonium salt? Allow a small sample of ammonium carbonate to stand on a watch glass over one laboratory period, and account for the change.

b. Add ammonium hydroxide solution one drop at a time to

freshly precipitated silver chloride and to copper sulfate solution. These reactions are probably due to the presence of ammonia, (NH_3) in the solution, and the soluble compounds formed are $\text{AgCl} \cdot 2\text{NH}_3$ and $\text{Cu}(\text{NH}_3)_4\text{SO}_4$.

50. Nitric Acid and the Oxides of Nitrogen.

a. Mix 10 grams of sodium nitrate (NaNO_3) with about 8 c.c. of concentrated sulfuric acid in a retort, arrange a receiving flask which can be cooled by running water, as in Exp. 35, and warm gently, protecting the retort with a wire gauze, until the nitric acid has been entirely distilled over. This should give about 5 c.c. of concentrated nitric acid. Note the color of the acid and of the gas above it. Pure nitric acid is colorless. Can you explain the color here? (If this acid cannot be used at once dilute it with one-half its volume of water before putting it away.)

b. Place about 1 c.c. of this acid in a test-tube, and dilute it with one-half its volume of distilled water. Moisten a piece of filter paper with this solution, then allow it to dry out on the radiator. Dilute a few drops of the acid with 2–3 c.c. of water and add barium chloride (BaCl_2) solution. Now add a small amount of sulfur to a new portion of the diluted acid, warm till reaction seems to cease, noting the appearance of the solution and of the gas evolved, and again test the solution with filter paper as before (see Exp. 44). Account for the difference in the two tests. What has become of the sulfur? Add a few drops of barium chloride solution to the mixture in the test-tube (after diluting with distilled water) and explain the change. Does nitric acid form a precipitate with barium chloride? What property of nitric acid does this reaction with sulfur illustrate?

c. Nitric Oxide.—Arrange a generating flask as usual, with thistle tube and delivery tube. Place about 3 grams of copper in the flask and add the rest of the nitric acid prepared above, diluted with one-half its volume of water. Heat the flask and contents and collect one bottle of the gas evolved by displace-

ment of water, noting the color of the gas in the generating flask, and in the gas collecting bottle. Account for the difference in color. At the same time collect a test-tube one-third full of the gas, note its color, and while the mouth of the tube is still under water pour into the tube (by pouring upward) the air from another test-tube which is two-thirds full of air, the rest of the tube being full of water and inverted in the bath like the first one. Account for the change in appearance and volumes of the gases after mixing. Shake the tube while still inverted under water to determine whether the gas formed is soluble in water. Does this experiment throw any light on the reason for the difference in color between the gas in the generating flask and in the collecting bottle? Write the reactions involved. Will the nitric oxide support the combustion of a burning splinter? Of phosphorus? (Test both of these on the bottle of gas.)

d. Nitrous Oxide.—Fit a small flask with stopper and delivery tube leading to the water trough. Place about 10 grams of ammonium nitrate (NH_4NO_3) in the flask and heat *gently* (too rapid heating may cause an explosion) collecting two bottles of the gas evolved by displacement of water. Note the color of the gas. Does it support the combustion of wood and phosphorus? Compare it with oxygen in this respect. Is there any apparent reaction with the oxygen of the air? Compare it with nitric oxide in this respect. How could a given sample of gas be tested to determine whether it was nitric oxide or nitrous oxide?

51. Behavior of Nitrates and Nitrites on Heating.

Heat samples of sodium nitrate, sodium nitrite and powdered lead nitrate in hard glass test-tubes. Note the color of the gas evolved and test it for oxygen. Add 1 c.c. of dilute hydrochloric acid to the residue after heating, in each case, again noting the color of the gas evolved. Explain the reactions and write equations. Arrange in tabular form the products obtained on heating sodium nitrate, ammonium nitrate, ammonium nitrite,

sodium nitrite and lead nitrate, and the products obtained when the residue from heating is treated with dilute hydrochloric acid.

The Combining Weight of a Metal by Oxidation with Nitric Acid.

Combining Weight of Tin. Quantitative.—Carefully clean and dry an evaporating dish and cover glass, weigh it, then place in it about 1 gram of tin foil and weigh again. Convert the metal to its oxide by adding dilute nitric acid (HNO_3), about 5 c.c. at a time, until all reaction apparently ceases. (Note the color of the gas evolved and explain.) Then remove the cover and evaporate on a steam bath to remove the excess of nitric acid and water, finally heating the covered evaporating dish with the direct flame to remove the last traces of water. Weigh, heat again and reweigh until the dish and contents have come to constant weight. The product in the dish is an oxide of tin. From the data obtained calculate the combining weight of the tin. If any nitrate of tin was formed during the process what has become of it? Was there any indication that a nitrate was being decomposed while the dish was heated with the direct flame? Write the reactions involved. The formula for the tin oxide may be determined from the combining proportions and the atomic weights of tin and oxygen.

CHAPTER IX

THE ATMOSPHERE

53. The Determination of Oxygen in the Air. Quantitative.

If a eudiometer is not available for this experiment a test-tube may be roughly graduated in 10 c.c., marking it with a pencil for writing on glass, or with file marks, and used in its place.

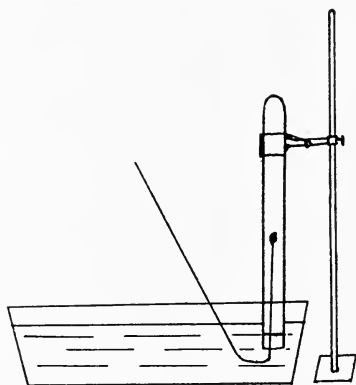


FIG. 15.

Invert a eudiometer containing the air of the laboratory over a water bath, clamp it in position, read the volume of air at atmospheric pressure, and insert into the air a bit of yellow phosphorus fastened on the end of a wire. Fig. 15. The phosphorus may be prepared by melting a piece of yellow phosphorus about the size of a pea, under water, placing the wire in the melted phosphorus and allowing the phosphorus to solidify in this position. *Yellow phosphorus should be kept under water and should not be handled with the bare hands, as it causes very unpleasant burns.* Allow this to stand until there is no further diminution in volume of the gas and again read the volume

at atmospheric pressure. Will the gas remaining in the tube support combustion? What is it? What is the per cent by volume of oxygen in the air? In this experiment the air was always saturated with moisture, and was measured at atmospheric temperature and pressure. Is it necessary to calculate the volume of the dry gas under standard conditions, before and after absorption of the oxygen, before calculating its per cent by volume? Would it be necessary to do so in order to calculate its per cent by weight?

54. The Composition of Air Dissolved in Water. Quantitative.

The composition of air dissolved in water may be very easily determined by the method used for the determination of oxygen in atmospheric air, the only change in manipulation being the source of the sample for analysis. Use the apparatus shown in Fig. 16, in which the eudiometer or graduated test-tube is fitted with a two hole stopper, one hole of which carries a tube leading just to the lower surface of a cork fitted to a 500 c.c. Erlenmeyer flask, while the other hole is fitted with a tube bent so that it leads under the surface of water in a beaker. Each of these connecting tubes must be in two pieces, connected with a rubber tube which can be closed with a pinch cock. Fill the whole apparatus completely with fresh tap water and clamp the eudiometer in an upright, inverted position above the Erlenmeyer flask, open both the pinch cocks (if water runs out on opening the cocks it is an indication of a leak, which must be corrected before proceeding) and slowly heat the water in the Erlenmeyer flask. The gas which separates will rise to the top of the eudiometer, while a small amount of water will run out into the beaker because of the expansion of the water on heating and its displacement by the gas. The water may be brought to the boiling-point, but it must not be allowed to boil lest steam escape, carrying with it some of the gases. If this does not supply a sufficient sample of gas for analysis the two pinch cocks may be closed, the Erlenmeyer flask refilled with fresh tap water, taking care that it is completely filled with water when the eudiometer is

replaced, and the procedure repeated until a sufficient sample is obtained. When a sufficient sample of the gas is obtained (20 c.c. will be enough for rough results) close both the pinch cocks, and bring the eudiometer, with its stopper and connecting tubes, under the surface of water in a water bath, then remove the stopper and read the volume after the gas has come to atmospheric temperature, and proceed to determine the oxygen as

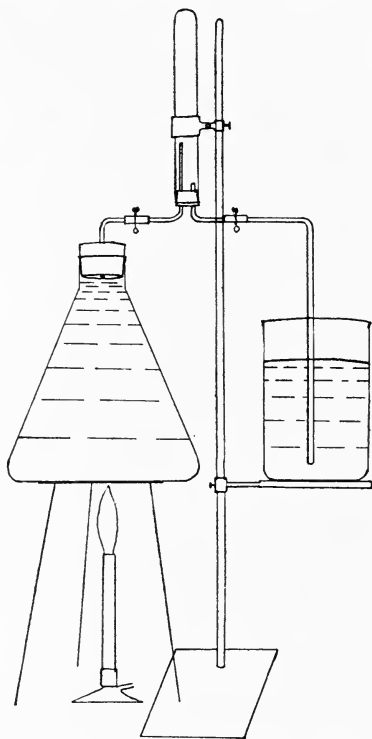


FIG. 16.

in Exp. 53. Calculate the per cent composition by volume of the gas analyzed, and compare with the per cent composition calculated from the solubility of the two gases and their partial pressures in the air. What would be the relative amounts dissolved if the air were 40 per cent oxygen? State Henry's law.

CHAPTER X

PHOSPHORUS, ARSENIC, ANTIMONY AND BISMUTH

55. The Oxides of Phosphorus.

Connect a hard glass combustion tube open at each end with a delivery tube fitted with a pinch cock and leading to the bottom of a test-tube or small flask one-third full of water which is fitted with a two hole stopper, the other hole of which carries a tube connected with a suction pump or aspirator bottle (Fig. 17).

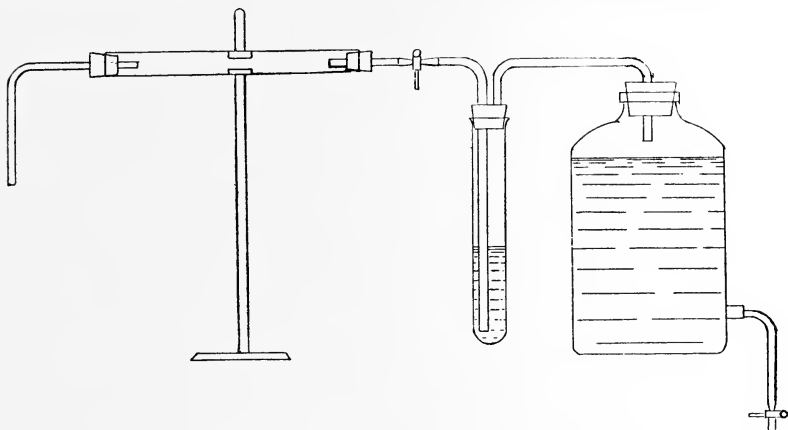


FIG. 17.

a. Place red phosphorus the size of a pea in the hard glass tube, light by touching with a red hot file, and by manipulating the pinch cock keep the phosphorus burning quietly while air is drawn over it and the products of combustion are absorbed by the water in the test-tube. Rinse out the combustion tube and the delivery tube into the solution in the test-tube, filter and test the filtrate with litmus. Make the following tests on this solution:

b. To a small portion add a few drops of potassium permanganate solution and warm.

c. To another portion add silver nitrate solution, then sodium bicarbonate (NaHCO_3) till a precipitate becomes permanent, then warm.

d. Add a small portion to albumin solution acidified with acetic acid. What do each of these tests indicate? What materials are thus shown to be present in the solution?

e. Evaporate the rest of this solution in a porcelain dish, adding nitric acid till brown fumes cease to be given off. (What are they? Explain their formation.) Evaporate till acid fumes are no longer given off, then heat with the direct flame until there is no further change, cool, and dissolve in water. Test this solution with potassium permanganate and silver nitrate solutions as above, and explain the results. What is now present in the solution? Allow the solution to stand for several days and test again with silver nitrate. Explain.

56. The Preparation of Primary, Secondary and Tertiary Orthophosphates, and their Decomposition Products on Heating. Quantitative.

a. Arrange twelve test-tubes in groups of three, and to the first of each group add a solution of primary sodium phosphate (NaH_2PO_4); to the second, secondary sodium phosphate (Na_2HPO_4); to the third, tertiary sodium phosphate (Na_3PO_4), and label each tube. To each of the tubes in the first group add a few drops of alizarine green, to the second group add phenolphthalein, to the third congo red, and to the fourth methyl orange. Record the colors formed in tabular form, noting at the same time the colors of each indicator in a solution of a strong acid and of a strong alkali. Which indicator should be used in the preparation of each of the sodium salts of orthophosphoric acid by neutralization of the acid with sodium hydroxide? Get the approval of an instructor on this point before proceeding.

b. Prepare 100 c.c. of an approximately formolar solution of phosphoric acid by diluting the concentrated acid (see page 7

for the table of per cent composition and density) and an equal amount of formalar sodium hydroxide solution from the solid sticks.

c. Into each of three evaporating dishes measure accurately (pipette) 10 c.c. of the acid solution after it has been well mixed, then neutralize the acid in each dish with the sodium hydroxide solution measured from a burette, and using the selected indicators, so that the primary, secondary and tertiary salts will be formed. Record the volumes of alkali required in each case. What ratio do these amounts bear to each other? What ratio should they bear, theoretically? Evaporate the solutions on the water bath, then heat the dry salts with the direct flame until all moisture is removed. What salt is now present in each case? Dissolve each of these three salts in water and test each solution with silver nitrate solution, and with albumin solution acidified with acetic acid. Do these tests confirm your conclusions regarding the salts present?

d. Devise and perform tests to show that most phosphates are insoluble in water but soluble in acids.

57. Arsenic.

a. Place about a gram of arsenopyrites in the center of a hard glass tube such as was used in preparing the oxides of phosphorus (Exp. 55), having one end closed with a cork, and the other carrying a small bore delivery tube. Heat the pyrite, when a sublimate will collect on either side of the hot part of the tube. What is it? After a considerable sublimate has collected allow the tube to cool, remove the residue of pyrite and connect the combustion tube with a test-tube or flask, and suction as in Exp. 55, Fig. 17, and burn one of the two deposits (save the other for *c*) collecting the products in the test-tube as before. Compare the solubility of arsenic oxides with that of phosphorus oxides. Filter the solution of the oxides (keep the insoluble residue for *b*) and test a small portion of the filtrate for sulfur dioxide by adding hydrochloric acid then barium chloride—a precipitate here would indicate what substance?

If any precipitate appears filter, then add potassium permanganate solution to the filtrate and heat. A precipitate here is due to what substance? Explain its presence.

b. Wash the residue of the arsenic oxide several times with distilled water, then punch a hole in the bottom of the filter paper and wash the oxide into a beaker with 20–30 c.c. of distilled water. Heat this to boiling to dissolve as much of the oxide as possible, filter if the solution is not clear and test the solution for sulfur dioxide as before. Is a precipitate of barium sulfate formed? Is the potassium permanganate decolorized? Explain. To a small portion of the solution add silver nitrate then sodium carbonate solution till a permanent precipitate forms (one drop is usually enough) and boil. Is the precipitate silver carbonate? Devise and perform an experiment to answer this question. What is the precipitate and what is its reaction on boiling? Compare with the similar compound of phosphorus.

Into the remainder of the solution pass hydrogen sulfide from a clean delivery tube (if the containers and solution have not been kept perfectly clean and free from electrolytes this will be a failure) and note the change in color. This should give a colloidal solution of arsenic sulfide. Compare the physical properties of this solution with those of a true solution. Divide the solution into two portions—to one add hydrochloric acid, to the other sodium chloride solution and explain the change. Would it be classified as a chemical or physical change?

c. Dissolve the other portion of the arsenic sublimate (*a*) in concentrated nitric acid and evaporate till the excess of acid is removed (hood), add water and to a small portion of the solution, filtered if not clear, add silver nitrate and sodium carbonate solutions till a permanent precipitate forms. Why is the sodium carbonate necessary? What is the precipitate? Boil. Compare with the silver arsenite precipitate prepared in *b*. Evaporate the rest of the solution to dryness and heat till all moisture is removed. What remains? Compare with the effect of heating phosphoric acid.

58. Antimony.

Treat 1-2 grams of stibnite (what is it?) with concentrated hydrochloric acid, warming in a covered evaporating dish until the reaction becomes slow. Note the odor of the gas evolved. What is it? Remove the solution from the excess of stibnite and add an equal volume of water. If a white precipitate forms (what is it?) add hydrochloric acid till it dissolves. If the solution turns yellow or a yellow precipitate appears (due to what?) boil till the color disappears. Make this solution alkaline with ammonium hydroxide, filter out the precipitate and wash with distilled water. What is the precipitate? Boil a small portion of the precipitate with distilled water and test with litmus. Is the hydroxide soluble? To two other portions of the precipitate add sodium hydroxide solution and hydrochloric acid respectively. Draw conclusions as to the acidic or basic character of the hydroxide. Compare with the character of the oxides of arsenic and phosphorus.

59. Bismuth.

Add 2-3 c.c. concentrated nitric acid to about 0.5 gram of metallic bismuth, boil the solution till nitric oxide fumes are no longer evolved and allow to crystallize by cooling and spontaneous evaporation. Heat a small portion of the crystals in a dry test-tube, noting the gas evolved. Add a second small portion to water and test with litmus. Account for the acid solution and for the insoluble substance. Add nitric acid to this until solution is complete, then prepare bismuth hydroxide by adding ammonium hydroxide to the solution. Filter and wash once with distilled water. Treat part of the precipitate with a 25 per cent solution of sodium hydroxide, and part with dilute hydrochloric acid. Is bismuth hydroxide basic or acidic? Indicate its ionization products and the effect of the addition of sodium hydroxide or hydrochloric acid on the ionization.

CHAPTER XI

CARBON

60. Carbon.

a. Fill a crucible about one-fourth full of cane sugar, and heat until the mass solidifies while hot, testing from time to time to determine whether the gas evolved burns. Heat 4-5 grams of copper oxide in a crucible to remove water, and when cool mix with some of the sugar charcoal in a dry, hard glass test-tube and heat, while watching for the condensation of moisture in the cool part of the tube. Pass some of the gases evolved into lime water, and explain the result. To what element in the charcoal is the moisture due? Is charcoal often pure carbon? Name the forms in which pure carbon occurs. Burn the rest of the charcoal, observing the rate and ease of combustion. Is carbon an active element? Does it burn with a flame?

b. Heat gently about 2 grams of soft coal in a crucible covered with an inverted funnel, noting the formation of a gas (does it burn?) and of tar. When combustible gases are no longer given off cool and observe the appearance of the coke formed. Is it pure carbon? To answer this apply the test used in *a* on one portion of the coke and place the crucible containing the rest of the coke in an inclined position and heat until the carbon has all burned away. What is left? What are some of the points to be considered in buying coal for heating purposes?

61. Carbon Dioxide.

Add dilute hydrochloric acid to marble in a generating flask (Fig. 7) and pass the gases evolved into lime water. What is formed? Attach a wash bottle to the generator so that the gas bubbles through water (to remove hydrochloric acid which might be carried over mechanically), then pass the gas into a

flask containing about 100 c.c. of water until the solution is saturated. Test the solution with litmus paper, taste it. What is present? Keep this solution (stoppered) for Exp. 64. Determine whether the gas is lighter or heavier than air, and fill a bottle of the gas by air displacement. Insert into this a burning taper. Pour the gas from one bottle into another and demonstrate that it has really passed from one to the other. Direct the stream of gas from the generator upon the flame from a burning match or taper, and explain. What commercial application is made of this property?

62. Determination of the Density of Carbon Dioxide. Quantitative.

Which of the methods previously used for density determinations can be applied to carbon dioxide? Arrange the apparatus and proceed with the determination after getting the approval of an instructor.

63. Carbon Monoxide. Poison. Do not Let the Gas Escape into the Room.

a. Mix about 3 grams of solid oxalic acid with an equal volume of concentrated sulfuric acid in a generating flask and heat, collecting three bottles of the gas by displacement of water. To stop the evolution of gas allow the generating flask to cool.

b. Add lime water to the first bottle and shake. What is formed?

c. Invert the second bottle as quickly as possible over a beaker of dilute potassium hydroxide solution and allow to stand until absorption is complete, then cover, set the bottle upright without pouring out the water that was absorbed and apply a light. What is this gas? What were the relative proportions of carbon dioxide and carbon monoxide in the gas mixture?

d. Apply a light to the third bottle of the gas mixture, and explain the result.

64. Hard Water.

a. Dilute some lime water with an equal volume of distilled water and pass in carbon dioxide until the precipitate first

formed (what is it?) redissolves (what is formed?). Filter the solution if it is not entirely clear, and divide into five portions. Boil one portion. To the other four add sodium carbonate, lime water, soap and alum solutions respectively. Draw conclusions as to the efficiency of these reagents and of boiling as softening agents?

b. Pour 20 c.c. of the carbonic acid solution prepared in Exp. 61 over a small amount of powdered marble or chalk, and allow to stand over night, then filter and test the filtrate by boiling, with sodium carbonate, etc. What was present in the solution? What is the importance of this reaction in nature?

65. Soap.

a. Dissolve about 0.5 gram of soap by heating in 25 c.c. of distilled water and test the solution with litmus and explain. Add solutions of sodium and calcium salts to different portions of this soap solution. Which would be more objectionable in water for laundry purposes, sodium or calcium bicarbonate?

b. Place equal quantities of animal charcoal in each of two test-tubes; to one add 10 c.c. of hot soap solution, to the other 10 c.c. of hot water, filter each and compare the filtrates. The soap solution tends to combine with the carbon to form an *adsorption* compound which reduces the tendency for the carbon to stick to other materials, here the filter paper. This property partly explains the action of soap in cleansing.

c. To 10 c.c. of soap solution add dilute hydrochloric acid to acid reaction. Warm gently until the white precipitate melts and floats on the surface. What is it?

66. Methane.

a. Grind together one part of sodium acetate with four parts of soda lime (quick lime and sodium hydroxide) and place the mixture in a hard glass test-tube fitted with a delivery tube. Incline the tube so that water driven from the contents on heating will not run back upon the hot part of the glass, and warm, collecting the gas evolved by displacement of water.

- b.* Shake one bottle of the gas with a few cubic centimeters of potassium permanganate solution.
- c.* Shake another with bromine water.
- d.* Burn a third and test the products of combustion remaining in the bottle for carbon dioxide.
- e.* Light a few bubbles of the gas as they escape from the water and note the nature of the flame.

67. Ethylene.

Add 9 c.c. alcohol slowly to 60 c.c. concentrated sulfuric acid, stirring constantly. Pour this mixture into a flask fitted with a delivery tube leading under water, and clamped so as to rest on a sand bath. Add a little sand to reduce foaming and heat gently until there is a steady evolution of gas (avoid overheating as the mixture foams considerably) and collect bottles of the gas, repeating all the tests applied to methane and comparing the results.

68. Acetylene.

a. Place 15 grams of calcium carbide in a flask fitted with a separatory funnel and delivery tube which is connected with a hard glass combustion tube open at each end and fitted with a delivery tube leading under water, Fig. 18. Generate the gas by allowing water from the separatory funnel to drop upon the calcium carbide, and collect bottles of the gas to which all the tests under methane should be applied. Compare with methane and ethylene.

b. After all the air has been driven out from the generating flask and delivery tubes heat the hard glass tube while still generating acetylene, and observe the deposit on the glass, and the changed appearance of the gas evolved, and explain.

69. The Oxidation Products of Alcohol.

Add 2 c.c. of concentrated sulfuric acid to a mixture of 8 c.c. water and 2 c.c. 50 per cent alcohol, mix thoroughly then add a small crystal of potassium dichromate and warm. Test the

gas evolved by holding in it strips of moist litmus paper, and note its odor. This is acetaldehyde.

To 1 to 2 c.c. of concentrated sulfuric acid add about 5 drops of 50 per cent alcohol, mix, then add a small crystal of potassium di-

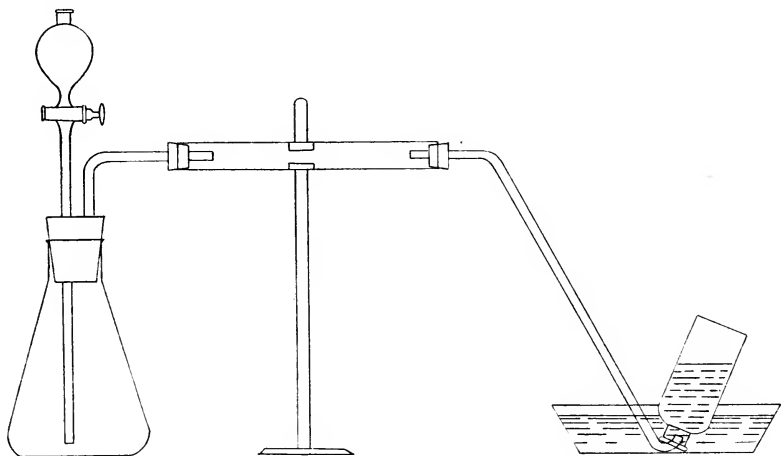


FIG. 18.

chromate and warm till the reaction begins (care!). Test the gas evolved with moist litmus paper and note its odor. What is it? Write equations for both reactions and explain the change in color of the solutions.

CHAPTER XII

SILICON

70. Silicon Tetrafluoride.

Grind together 1 gram of silicon dioxide (sand) and 3 grams of calcium fluoride, moisten with concentrated sulfuric acid and heat in a test-tube fitted with a delivery tube leading just above the surface of water. What is the precipitate formed on the surface of the water? Explain the fumes above the water. Test the water solution by filtering, then adding to the filtrate dilute hydrochloric acid and barium chloride solution, and warming. What is formed? Write all reactions.

71. Silicic Acid.

a. Grind together in an iron crucible 1 gram of sand and 3 grams of sodium hydroxide (solid) and heat until the mass which at first fuses, turns solid. Cool, dissolve in 25 c.c. water and filter. To one portion of the filtrate (what is it?) add concentrated or dilute hydrochloric acid very slowly. Pour another portion, about 6 c.c., into a test-tube containing about 6 c.c. of concentrated hydrochloric acid. Has there been any difference in the chemical reaction in these two tubes? Explain the difference in appearance. Evaporate part of the clear solution of silicic acid in a porcelain dish and heat the dry residue with the direct flame, then cool and determine whether it is still completely soluble in water.

b. Dialyze the rest of the silicic acid solution as follows: Prepare a collodion sack for dialysis by wetting the inside walls of a 50 c.c. beaker which has been carefully cleaned and dried, with a solution of collodion in ether and allowing the ether to evaporate, then when the odor of ether has disappeared remove the sack from the beaker (loosen the edge of the collodion from

the glass with a knife, then the sack may be withdrawn with the finger, or by allowing water to run in between the glass and the collodion) and fill with the solution of colloidal silicic acid. Suspend this sack in a beaker of distilled water so that the level of the water is slightly lower than the level of the solution in the sack (Fig. 19) and change the dialysate (the solution in the

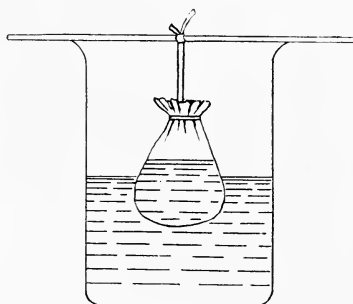


FIG. 19.

beaker, consisting of materials which have dialyzed through the membrane) frequently until it gives only a very faint test for chlorides after standing in contact with the sack for about an hour. Test portions of the dialyzed solution (the solution remaining in the sack) with barium chloride and copper sulfate solutions. Would barium or copper silicates be soluble if formed? Is the silicic acid ionized in this solution? Evaporate another portion to dryness, heat with the direct flame and determine whether the residue is soluble in water. Has it undergone a chemical change?

CHAPTER XIII

BORON

72. Preparation of Boric Acid from Borax.

Saturate 50 c.c. of distilled water with borax, test the solution with litmus and account for the result. Add dilute sulfuric acid to this solution until no further precipitate forms, then cool and filter the cold solution. Wash once or twice with cold distilled water then purify the precipitate by redissolving in the least possible amount of boiling water and cool under running water. Filter and wash the precipitate with cold water till the wash water is free from sulfates, then dry thoroughly by pressing between folds of filter paper and then warming on the radiator or in a drying oven at a temperature below 100°C . What is the residue? Dissolve some in water and test the solution with litmus. Mix a small portion with an equal quantity of sodium chloride and heat the mixture in a test-tube. What is formed? Heat a small portion in a dry test-tube. Do these tests prove whether the material is an acid or an acid anhydride?

73. Boric Acid Anhydride or Boron Trioxide from the Acid. Quantitative.

Carefully clean and weigh a porcelain crucible and place in it about 1 gram of the well dried acid from the previous experiment (Exp. 72) and weigh accurately. Cover the crucible and heat, at first gently and then with the full heat of a bunsen burner. Cool and weigh, and repeat until the crucible and contents come to constant weight. Calculate the per cent of water in the acid, and the number of molecules of water to one of the acid anhydride.

74. The Preparation of Metallic Boron.

Heat some of the prepared boric acid (Exp. 72) in a porcelain crucible with the direct flame till all water is removed, then mix

about 1 gram of the anhydride with 1.5 grams of magnesium powder. Place a small portion of this mixture in a crucible and heat till glowing ceases, then add gradually the rest of the mixture, heating continuously. Grind the product in a mortar, wash it with dilute hydrochloric acid then with hot concentrated hydrochloric acid diluted 1:1 with water, and finally with distilled water until the filtrate comes through brown, due to the formation of a colloidal solution of boron, and indicating that electrolytes are not present in appreciable amounts. Test the solubility of this boron in concentrated nitric, hydrochloric and sulfuric acids. Fuse a small portion with an equal quantity of solid potassium hydroxide. What is formed? Dissolve the fused mass in as little water as possible and acidify with dilute sulfuric acid. What is formed? How may tertiary sodium borate (Na_3BO_3) be prepared? Is it stable in solution?

75. Borates of the Heavy Metals.

Will borax or boric acid precipitate copper borate from copper sulfate solution? Is copper borate soluble in water? To answer this question prepare a borax bead by fusing borax on the loop of a platinum wire, dip the bead into copper oxide and heat until a transparent colored bead is formed. If the bead does not become clear after heating several moments it is because too much copper oxide has been added. What copper compound is present in the bead? Break up the bead and test its solubility in water. Boil the water suspension and note the change in color of the precipitate, which is due to the formation of copper oxide from the hydroxide first formed. Explain the presence of copper hydroxide on treating the bead with water, on the basis of the ionization theory.

CHAPTER XIV

METALS AND NON-METALS

76. Hydrolysis.

Dissolve crystals of sodium chloride, potassium chloride, ammonium chloride, cupric chloride, ferric chloride, stannous chloride and phosphorus pentachloride each in a separate beaker in about 5 to 10 c.c. of water. Test the solutions with litmus, then boil each, covering the beaker with a watch glass on which is placed moist red and blue litmus paper so that it will come in contact with the vapor from the boiling solution. Note and account for the effect of the vapor on the litmus in each case, then allow the solutions to evaporate to dryness on the water bath, dissolve the *dry* residue in very dilute nitric acid and test the solution for chlorides. Which of these salts have undergone no hydrolysis? Which have been partially hydrolyzed? Which have been completely hydrolyzed? Define hydrolysis. What is the effect of water on the chlorides of metals? Of non-metals? Do the results of this experiment lead to the conclusion that there is a sharp distinction between metals and non-metals?

77. Ionization.

a. Make up 100 c.c. each of thrice normal solutions of sulfuric, hydrochloric, acetic, phosphoric and oxalic acids. To determine the relative strength, or degree of ionization of these acids place 5 c.c. portions of each acid in separate test-tubes, label each tube and place them all in a beaker containing water, so that they will not suffer any great change of temperature. Now into each tube drop a small granule of zinc and note the rate of the reaction, judging by the rate of evolution of hydrogen in each tube. Name the acids in the order of their

strength, then heat the water bath till it reaches about 50° and again note the rate of the reaction, and record. If the weakest reaction were considered the standard or unit of strength, what values would you give the other acids? Compare your values with those in the table of degrees of ionization. Repeat this experiment using small chunks of marble instead of zinc, and arrange the acids according to their strength, as before. How does this order agree with that arranged with reference to their action on zinc?

b. Make a saturated solution of potassium chromate, and place *equal quantities* in each of six test-tubes. Now add to one of the test-tubes, drop by drop, the acid found to be weakest in the previous test, until a just noticeable change in color has occurred, then add to each of the other test-tubes an equal number of drops of each of the acids, label each, and to the sixth tube add an equal number of drops of concentrated sulfuric acid (this represents the greatest possible change). Shake each tube and arrange in the order of their depth of color, and compare with the order of the acids with respect to their activity on zinc or marble.

c. Repeat this procedure, using a solution of 6 grams potassium iodide and 1 gram potassium bromate per liter instead of the solution of potassium chromate. In this case each of the tests will finally come to the same color, but the weakest acid will bring about the change most slowly, so that they may be arranged according to either of two variables, *i.e.*, the depth of color at a fixed time after adding the acid, or the length of time required for each tube to come to a definite depth of color. Record the acids in the order of one of these variables, and compare with the order found in *a* and *b*.

78. Indicators.

a. Dilute 1 c.c. of concentrated hydrochloric acid with 9 c.c. of water, mix thoroughly and add *one drop* of this diluted acid to 100 c.c. of distilled water (A). Mix thoroughly and add equal portions to four test-tubes, then add to each test-tube a

few drops of one of the indicators, methyl orange, methyl red, congo red and litmus, the acid and alkali colors of each of which should be previously determined. Dilute 50 c.c. of this acid (A) with 50 c.c. of distilled water, mix thoroughly and test again with each of the four indicators, and repeat this diluting and testing until each of the four indicators have changed color. Which indicator changes color in the greatest hydrogen ion concentration? which in the least?

b. Make up a solution of ammonium hydroxide in exactly the same way that the hydrochloric acid solution was prepared, using the concentrated ammonium hydroxide, sp. gr. 0.90, and test the successive dilutions of this with phenolphthalein, tropaeolin O and litmus. Which indicator changes color in the greatest hydroxyl ion concentration? which in the least?

c. To two portions of 10 c.c. of normal sodium acetate add methyl orange and phenolphthalein respectively as indicators (is the reaction of sodium acetate acid or alkaline?) then titrate each with normal acetic acid and record the quantities of acid used to neutralize the solution, with each indicator. Explain why these quantities should be different. Now repeat the procedure titrating with normal hydrochloric acid instead of acetic, record the quantities used and explain the different effects of the two acids. In neutralizing sodium hydroxide with acetic acid which of these two indicators should be used? In neutralizing with hydrochloric acid would it make much difference which was used?

d. Test a solution of ammonium sulfate with methyl orange and phenolphthalein, and titrate equal volumes of the solution with normal ammonium hydroxide or normal sulfuric acid, according as the original solution reacts acid or alkaline, using these two indicators. What indicator should be used in neutralizing a strong acid, as sulfuric acid, with a weak base, as ammonium hydroxide? Titrate equal quantities of normal ammonium hydroxide with normal acetic acid, using methyl orange and phenolphthalein as indicators. Will these indicators serve to show the neutral point in this case? Why?

79. Indicators. Quantitative.

a. By titrating a mixture of sodium hydroxide and sodium bicarbonate with normal hydrochloric acid it is possible to titrate only the sodium hydroxide, if the indicator is chosen so that as soon as carbonic acid is formed it shows acid reaction, and with another indicator which is not affected by so low a hydrogen ion concentration as is supplied by carbonic acid, but is affected as soon as free hydrochloric acid exists in the solution, it is possible to determine the total sodium hydroxide plus sodium bicarbonate in the solution. Which indicator should be used for each titration? Test your decision by trying a solution of sodium bicarbonate alone with the first indicator, and determine how much normal hydrochloric acid is required to give acid reaction, and by trying the second indicator to determine that carbonic acid does not give acid reaction, but that free hydrochloric acid does.

b. Titrate equal volumes (pipette) of a solution of these two salts with normal hydrochloric acid, using the two indicators chosen, and from these two titrations calculate the amounts of sodium hydroxide and sodium bicarbonate present in the mixture. In the original solution could sodium hydroxide and sodium bicarbonate exist together? What would be formed? Is there an indicator which would give an acid reaction with sodium bicarbonate, so that when a mixture of sodium hydroxide and sodium carbonate were titrated only the sodium hydroxide would be neutralized? Write all the reactions which occur when sodium hydroxide and sodium bicarbonate are mixed in solution, and when the mixture is titrated, first with one indicator and then with the other. From the results of the titration above calculate the amount of sodium carbonate in the given mixture.

CHAPTER XV

THE ALKALI METALS

80. The Transition of Sodium Sulfate from the Anhydrous to the Hydrated Salt.

Add 30 grams of anhydrous sodium sulfate (if only the hydrated salt is available calculate the quantity required to give 30 grams of the anhydrous salt) to 40 c.c. of water heated to 50° C., and hold the mixture at that temperature for about five minutes. Is the solid phase the hydrated or the anhydrous salt? Can you suggest an experimental proof for your answer to this question? Now cool by immersing the beaker in water at 15° to 20° C. and record the temperature of the solution every half minute for ten minutes, stirring it constantly. If the temperature falls below 25° drop in a few crystals of the hydrated salt. Make a plot to represent the change in temperature with the time, letting the horizontal axis represent temperature and the vertical axis time, and explain the sudden break in the curve. Is the change from the hydrated to the anhydrous salt endothermic or exothermic?

81. Sodium Carbonate by the Solvay or Ammonia Process.

(Adapted from Blanchard's Synthetic Inorganic Chemistry.)

a. To 50 c.c. of concentrated ammonium hydroxide (0.90 specific gravity) add 150 c.c. of distilled water. Place in a flask and add 60 grams of sodium chloride free from lumps. Shake until the salt is nearly or quite dissolved, and filter the solution, if it is not perfectly clear. Pass a delivery tube through one hole of a two-hole, tightly fitting stopper placed in a 300 c.c. flask. Provide a plug for the other hole. Let the tube dip into the solution which is placed in the flask, and pass in carbon dioxide gas from a Kipp generator until all the air has

been displaced from the flask; then close the flask and allow the gas to pass in as fast as it will be absorbed. What is formed? Occasionally as the action seems to slacken, loosen the plug for a moment. Shake the flask frequently. It will take several hours for the solution to absorb sufficient carbon dioxide, and it may be left over night connected with the generator. When no more gas can be absorbed pour the mixture from the flask onto a fluted filter paper, wash the precipitate with a small amount of cold water, and allow it to dry in the air. Look up the solubilities of the substances started with and the final products of this reaction and explain upon what the success of the process depends. Why could not ammonium chloride be used instead of ammonium hydroxide? What remains in the filtrate from the bicarbonate? Would the process be financially a success if this filtrate was discarded? How may the ammonium hydroxide be regenerated from it and what is the final waste product?

b. Add hydrochloric acid to some of the solid bicarbonate prepared above. Heat a small portion and test the gas evolved for carbon dioxide. To the residue after heating add hydrochloric acid. What is the reaction on heating?

c. Heat about 1 gram of the bicarbonate till decomposition is complete, dissolve the product in very little water, test with litmus and compare with the effect of sodium bicarbonate solution on litmus. Add a solution of lime water (calcium hydroxide) till there is no further precipitation, and filter the precipitate? What is in solution?

82. Potassium Nitrate from Sodium Nitrate and Potassium Chloride. (Adapted from Blanchard's Synthetic Inorganic Chemistry.)

Dissolve 50 grams of sodium nitrate and 44 grams of potassium chloride in 100 c.c. of water and evaporate on the water bath in a porcelain dish, adding the solution in portions if the dish will not hold it all at once, till the solution is half its original volume. Without letting the liquor cool separate it

from the crystals which have formed during the evaporation. This may be done by decantation or by filtering through a fluted filter paper which has been wet with hot water. Allow the filtrate to cool by holding the beaker containing it under running water, and filter out the second crop of crystals. What four salts may be present in the solution after potassium chloride and sodium nitrate have been mixed together? Look up the solubilities of each of these salts and determine which would separate out first from a solution containing equi-molecular amounts of each, on evaporating at 100° . Test the first crop of crystals to determine if your reasoning is correct. If half the sodium and chloride ions in the original mixture were removed, what would be left in the solution and in what proportions? Considering their solubilities at high and low temperature, and the relative amounts present, which salt would you expect would separate out on cooling the solution? Devise and apply a test on the second crop of crystals to determine whether your conclusion is correct. Why is potassium nitrate used for the manufacture of gunpowder rather than sodium nitrate?

CHAPTER XVI

COPPER AND SILVER

83. Copper and Cupric Compounds.

a. Treat bright copper filings with dilute sulfuric and dilute hydrochloric acids. Does the metal dissolve in either case? Pour off the dilute acids and replace them with concentrated acids, warm, and note the effect in each case. Test the effect of both dilute and concentrated nitric acid on copper and account for the effect of each acid on the copper. Will copper displace hydrogen from an acid?

b. Heat some copper filings in an open crucible and note the change in appearance. Test the product for solubility in dilute sulfuric and dilute hydrochloric acids, and account for the difference in behavior of copper before and after heating in air.

c. Add ammonium hydroxide drop by drop to about 5 c.c. of copper sulfate solution, shaking after each addition, until the mixture smells strongly of ammonia. Account for every change which occurs. To another portion of copper sulfate solution add sodium hydroxide solution till the mixture is alkaline, then heat, and account for every change which occurs.

84. Cuprous Compounds.

a. Prepare a solution of 3.5 grams of copper sulfate in 50 c.c. of water, and another solution of 17 grams of Rochelle salt (sodium potassium tartrate) and 7 grams of potassium hydroxide (or its equivalent of sodium hydroxide) in 50 c.c. of water. Mix these solutions and heat to boiling then add a solution of glucose until all the copper is precipitated. How can that point be recognized? Filter and wash the precipitate (what is it?) with warm water several times, then allow to dry.

b. To a small portion of the precipitate add dilute hydrochloric acid. What is formed? Test different portions of this

white precipitate for solubility in concentrated hydrochloric acid and in ammonium hydroxide. Is the cupric ion present in either of these solutions? State the reason for your answer. Pour the concentrated hydrochloric acid solution into an excess of water and explain.

c. Add ammonium hydroxide to a small portion of the red cuprous oxide and explain the color changes.

d. Add dilute sulfuric acid to another portion of the cuprous oxide. Note the color of the solution and of the insoluble residue. Devise and apply tests to prove that this residue is not cuprous oxide, but is metallic copper. Is the cuprous or cupric ion present in the solution? What is the oxidizing and what the reducing agent in this reaction?

e. To a few cubic centimeters of copper sulfate solution add an excess of potassium iodide solution, filter and wash the precipitate first with a little potassium iodide solution (why?), then with water. Explain the color of the filtrate. To one portion of the precipitate add dilute potassium hydroxide and warm. Is this the oxide of cupric or cuprous copper? To another portion of the precipitate add ammonium hydroxide. What is formed?

85. Electromotive Series.

Place the following mixtures in test-tubes, taking especial pains to see that the metal has a bright, shiny surface, *i.e.*, is not covered with an oxide. Observe the changes occurring, and complete the chemical equations

Cu + ZnSO₄ solution.

Pb + ZnSO₄ solution.

Cu + Pb(NO₃)₂ solution.

Pb + Cu(NO₃)₂ solution.

Cu + Hg(NO₃)₂ solution.

Pb + Hg(NO₃)₂ solution.

Hg + ZnSO₄ solution.

Hg + Pb(NO₃)₂ solution.

Hg + CuSO₄ solution.

Zn + Pb(NO₃)₂ solution.

Pb + HCl (dilute).

Zn + Cu(NO₃)₂ solution.

Zn + Hg(NO₃)₂ solution.

These reactions are not instantaneous and should be allowed to continue during the time of at least one laboratory period. Arrange the four elements in the order of their "solution tension" or tendency to change from the free to the ionic condition. Refer to the last part of Exp. 13 and find the place for hydrogen in the series.

86. Silver Oxide.

a. Prepare silver oxide by adding an excess of potassium hydroxide to about 20 c.c. of silver nitrate solution, filter out the oxide and wash, then test small portions of the precipitate for solubility in ammonium hydroxide and dilute sulfuric acid. Heat the remainder of the precipitate in a porcelain crucible, and note the change in appearance and test small portions for solubility in ammonium hydroxide and dilute sulfuric acid. Explain the difference in conduct before and after heating. Dissolve the remaining silver in concentrated nitric acid which has been diluted with an equal volume of water, avoiding an excess of the acid, then evaporate the solution till crystals begin to form on the surface, then crystallize by cooling and spontaneous evaporation.

b. Convert a small portion of the silver nitrate into silver chloride, add ammonium hydroxide and explain. Devise and apply a method for converting the rest to silver sulfate. Compare the properties of the cuprous and silver ions.

87. Silver Chloride Emulsion.

Prepare a 1 per cent gelatin solution (gelatin will dissolve readily on warming with water) and to a measured amount of the solution (about 25 c.c.) add two drops of concentrated hydrochloric acid and an excess of silver nitrate solution and compare with the appearance of the silver chloride when the same procedure is followed, using distilled water instead of the gelatin solution. Allow the two preparations to stand for an hour or more and again compare. What is the technical importance of silver chloride emulsions?

CHAPTER XVII

CALCIUM, STRONTIUM AND BARIUM

88. Calcium Carbonate and Lime.

a. Heat about 2 grams of well ground marble in a hard glass test-tube fitted with a stopper and delivery tube, and test the gas evolved by passing it through lime water. Remove small portions of the material in the test-tube at intervals of about fifteen minutes, and test with hydrochloric acid to determine the rate of decomposition. How could marble be kept from decomposing at 800° ? After the heating has been continued for about an hour test a portion of the residue in the test-tube for solubility in water and for the reaction of the solution toward litmus, and compare with the original marble. Account for the differences noted. Filter out the insoluble residue and pass carbon dioxide into the filtrate. What is formed? Add a little quicklime to water and note the temperature change, and the reaction of the solution toward litmus.

89. The Relative Solubility of the Hydroxides of Calcium, Strontium and Barium. Quantitative.

a. Make saturated solutions of barium, strontium and calcium hydroxides by adding an excess of the solid to 50 c.c. of distilled water and allowing to stand in stoppered flasks until the supernatant liquid is clear. (Keep these solutions for Exp. 90.) Now make 200 c.c. of an approximately 0.1 normal hydrochloric acid solution by diluting the concentrated acid (sp. gr. 1.16). Take 10 c.c. of each of the hydroxide solutions, measured with a pipette, from the clear supernatant liquid. Neutralize these with the acid from a burette, recording the exact quantity of acid required in each case and using an indicator which will not give the acid color in so slight a hydrogen ion concentration

as comes from carbonic acid. Why? Calculate the relative solubilities of these three hydroxides. If the acid is assumed to be exactly 0.1 normal what are the absolute solubilities?

b. Titrate 100 c.c. of tap water in the same way. What is the alkaline substance? Assuming that the alkali in the tap water is lime, express the concentration of the tap water in terms of a saturated solution of lime. (That is, if a saturated solution was taken as unity, this would be some fraction of a saturated solution.)

90. The Relative Solubilities of Calcium, Strontium and Barium Bicarbonates.

Measure out 10 c.c. of each of the saturated solutions of calcium, strontium and barium hydroxides prepared in Exp. 89 and pass carbon dioxide from a Kipp generator into each of these solutions, adding water to each gradually, while the carbon dioxide continues to pass through, until the precipitate first formed just dissolves in the excess of water saturated with carbon dioxide. (A small granular precipitate in the case of the barium may be ignored.) Now measure the total volumes of each of the solutions, and calculate the relative solubilities of the bicarbonates from that of the hydroxides (Exp. 89) and these measured volumes.

91. The Relative Solubilities of Calcium, Strontium and Barium Sulfates.

Prepare calcium sulfate from a small piece of marble (can this be done by adding sulfuric acid directly? Why?). Filter and wash the calcium sulfate free from sulfuric acid (would you test this with litmus or with barium chloride? Why?) then make a saturated solution of this calcium sulfate and add the clear solution to strontium chloride solution and warm. What is precipitated? What remains in solution? Add the filtrate from this to barium chloride solution and explain. Draw conclusions regarding the relative solubility of barium, strontium and calcium sulfates. Explain why calcium sulfate precipitates from solutions of calcium chloride and sulfuric acid more

slowly than barium sulfate from solutions of barium chloride and sulfuric acid.

92. Preparation and Properties of Calcium Chloride.

a. Prepare a solution of calcium chloride from about 25 grams of marble, taking care to avoid an excess of hydrochloric acid, filter and crystallize by boiling until crystals begin to separate, then allow to cool, remove the crystals from the mother liquor, and dry them on filter paper. Boil down the mother liquor and continue heating, with stirring, until the solid residue has become quite white and opaque. What is the difference between these two preparations? How much of the crystallized salt would be equivalent to a gram of the anhydrous?

b. Add equivalent quantities of each of these salts (finely ground) to 10 c.c. of water, taking the temperature before addition and after solution of the salt, and account for the difference.

c. Repeat *b*, using equal weights of finely chopped ice or snow instead of water, and account for the difference in effect of the two salts, and for the difference in effect as compared with the previous test with water. Allow samples of each of the preparations to stand in the open air and account for the change.

CHAPTER XVIII

MAGNESIUM ZINC AND MERCURY

93. The Equivalent Weight of Magnesium. Quantitative.

Prepare an apparatus as shown in Fig. 20, in which an ordinary test-tube is fitted with a two hole stopper, one hole of

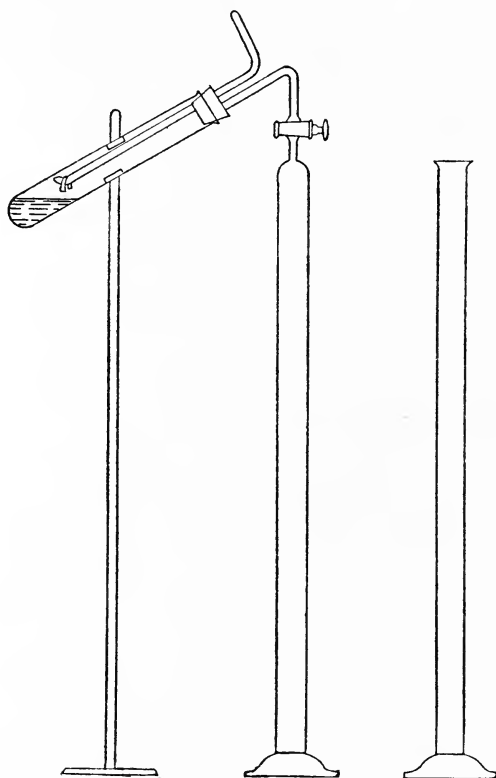


FIG. 20

which carries a glass rod which has been bent over just a little at its lower end, and after being put into the cork has been bent

at right angles at the other end, while the other hole carries a tube bent at an acute angle, to connect with the gas burette. Make the apparatus air tight, then fill the burette to the zero mark with water, place about 10 c.c. of dilute hydrochloric acid in the test-tube, and hang on the glass rod a weighed quantity of magnesium ribbon (not more than 0.05 gram) or a measured length, the weight of which can be calculated from a factor, weight per centimeter, on the bottle. Connect the apparatus, open the burette cock and read the volume (at atmospheric pressure), drop the magnesium ribbon into the acid by turning the glass rod, and after the magnesium is all dissolved read the volume again, noting the temperature of the room and the atmospheric pressure. Calculate the weight of hydrogen evolved, and the equivalent weight of magnesium.

94. Magnesium Carbonate and Oxide.

Heat magnesium carbonate as calcium carbonate was heated (Exp. 88), testing a small portion with hydrochloric acid every ten minutes till decomposition is complete, add water and test the suspension with litmus. Compare these with the same experiments on calcium. Add a saturated solution of ammonium chloride to the suspension of magnesium oxide, test again with litmus and account for the changes.

95. The Properties of Metallic Zinc.

Heat a granule of zinc in an open crucible, noting and explaining all changes which take place during the heating and subsequent cooling. Compare with the conduct of silver under similar conditions. Tabulate the melting-points of the metals of the first and second groups of the periodic system and formulate a statement of the relation of melting-points to atomic weights.

96. The Equivalent Weight of Zinc. Quantitative.

Repeat the manipulation described in Exp. 93 using about 0.1 gram of zinc, and place in the test-tube a small piece of platinum wire with which the zinc may come in contact, to hasten its solution. Calculate the equivalent weight of zinc.

97. Preparation of Zinc Sulfate.

a. Make a suspension of 5 grams of finely powdered zinc oxide in water, test this with litmus, and dissolve by adding acetic acid, or dissolve in dilute hydrochloric acid and add sodium acetate. Explain why these two procedures would give the same hydrogen ion concentration. Place this solution in an erlenmeyer flask fitted with a one hole stopper carrying a delivery tube projecting below the surface of the liquid, and pass in hydrogen sulfide with the stopper loose until the air in the flask has been displaced, then close the flask, when hydrogen sulfide will be supplied as fast as absorbed, and allow the flask to stand until the solution is saturated. What is the precipitate? Filter and wash, then dry the moist precipitate in an evaporating dish, stirring constantly, and when entirely dry heat in an open crucible to dull red for half an hour, or until a small test portion shows the presence of appreciable amounts of a soluble sulfate. Account for the formation of a sulfate. Write the equation. Dissolve in water, warm and filter. Test the filtrate with litmus and explain, then evaporate the solution to crystallization. Devise and apply a test to determine whether the insoluble residue is mostly zinc oxide or zinc sulfide.

b. Dissolve the zinc sulfate prepared in *a*, acidify with dilute sulfuric acid and pass hydrogen sulfide into a portion of the solution, then add an equal volume of sodium acetate solution and again pass in hydrogen sulfide. Account for the difference.

c. To two other portions of the zinc sulfate solution add sodium hydroxide till a permanent precipitate forms, then to one add an excess of sodium hydroxide, and to the other hydrochloric or sulfuric acid. Is zinc hydroxide a base or an acid? Indicate its products of ionization and the effect which the presence of a base or an acid has upon its ionization.

98. The Preparation and Properties of Mercurous and Mercuric Nitrate.

a. Clean 5 grams of mercury by pouring through a dry filter paper through which a pin hole has been pricked at the apex.

Calculate the amount of concentrated nitric acid required to convert this to mercurous nitrate (HgNO_3) and take a little less (why?) than the calculated amount of the concentrated acid (specific gravity 1.40,) and dilute with twice its volume of water. Dissolve the mercury in this, warming gently when necessary to keep the reaction going, and after it has apparently ceased (a good indication of complete reaction is the formation of a yellow precipitate on diluting a small portion of the solution with an equal volume of water—explain this test after the experiment has been completed) pour off the solution from the residue of undissolved mercury, cool and allow to crystallize. To separate portions of the mother liquor add hydrochloric and sulfuric acids and ammonium hydroxide. Compare the solubility of mercurous nitrate, sulfate and chloride. Add water to some of the crystals, then warm and test the water solution with litmus. Account for the test. What is the precipitate? Is it soluble in water? In nitric acid?

b. Dissolve the rest of the mercurous nitrate in as little dilute nitric acid as possible and warm, adding concentrated nitric acid as necessary till brown fumes are no longer given off. What reaction is taking place? Calculate the amount of concentrated nitric acid required to convert 5 grams of mercury to mercuric nitrate ($\text{Hg}(\text{NO}_3)_2$) and compare with the quantity required for mercurous nitrate. Crystallize this salt. How does its solubility compare with that of mercurous nitrate? Dissolve some of the crystals in water, adding dilute nitric acid if necessary, and test the solution with hydrochloric and sulfuric acids and ammonium hydroxide, and compare with the mercurous compounds. Does the ammonium hydroxide form mercuric hydroxide? Devise and apply a test to determine this. Add some bright metallic copper to the mercuric nitrate solution. Where is mercury in the electromotive series? What metals will be precipitated from solution by mercury? Add a small amount of zinc to mercuric nitrate solution and warm till the zinc is nearly all dissolved, then test the solution by adding hydrochloric acid. What was the effect of the zinc?

CHAPTER XIX

ALUMINIUM

99. Aluminates and Aluminium Hydroxide.

Dissolve about 2 grams of aluminium in dilute potassium hydroxide, avoiding an excess of the alkali. What remains in solution? What gas is evolved? Filter the solution and pass hydrogen sulfide into a small portion. Is a sulfide precipitated? Prove your answer experimentally. Pass carbon dioxide into the main portion of the solution. What is the precipitate? Filter and wash, and show experimentally that it is not a carbonate. Test small portions of this precipitate for solubility in acids and alkalis, and explain in terms of the ionization theory. Dissolve the main portion of the precipitate in hydrochloric acid, evaporate until crystals appear on the surface of the liquid, which should be kept strongly acid, and crystallize by cooling and spontaneous evaporation. Dry the crystals, dissolve a small portion in water and pass in hydrogen sulfide. Compare with the action of hydrogen sulfide on the alkaline solution and explain. Heat the rest of the crystals in a dry test-tube till decomposition is complete, noting the odor of the gas given off. What is it? Is the residue a chloride? Test this experimentally.

100. Water of Crystallization in Alum. Quantitative.

Calculate the quantities of aluminium sulfate (hydrated) and potassium sulfate necessary to make 5 grams of the crystallized aluminium alum, and dissolve these amounts in 20 c.c. warm water, cool and allow to crystallize slowly. Dry the crystals on filter paper, then by standing in the open air, and after they are thoroughly dry determine the water of crystallization by placing about 1 gram in a clean, dry crucible which has been

previously weighed, and weighing accurately. Cover this crucible and place in another crucible of the same size and heat with a flame not more than two inches high, and at least two inches from the bottom of the lower crucible. After most of the water has been driven off the lower crucible may be removed and the small flame brought just below, but not quite touching the crucible containing the alum, and the heating continued until constant weight is attained. Too high a temperature will cause decomposition of the aluminium sulfate. Calculate the per cent of water in alum from this experiment, and from the formula, and compare.

101. Aluminium in Kaolin.

Fuse some potassium pyrosulfate in a crucible and add powdered kaolin in small portions until the mass will no longer melt, then cool, dissolve in boiling water, filter and add ammonium hydroxide to the filtrate. What is formed? Test its solubility in potassium hydroxide and in hydrochloric acid.

CHAPTER XX

LEAD AND TIN

102. The Properties of Metallic Lead and of Lead Salts.

a. Test the solubility of metallic lead in hydrochloric, acetic, sulfuric and nitric acids. To solutions of lead nitrate add each of the above acids, first cold, then warm. From the information so gained explain the effects of the acids on metallic lead. From the position of lead in the electromotive series would you expect it to dissolve in these acids?

b. Add sodium hydroxide solution to lead nitrate solution, first in small amount then in excess, and pass hydrogen sulfide into the alkaline solution. Devise and apply a test to determine whether the sulfide or hydroxide is precipitated here. Pass hydrogen sulfide into a hot solution of lead acetate, acidified with hydrochloric acid. Is the sulfide or hydroxide precipitated? Compare these results with those obtained on similar treatment of aluminium. Which element shows the more basic properties? From the positions of lead and aluminium in the Periodic System which would you expect to show most basic properties?

103. The Oxides of Lead.

a. Heat a small sample of red lead in a dry test-tube, noting the change in appearance, and at the same time test for oxygen. After decomposition is complete divide the residue into two portions; to one add dilute nitric acid and to the other sodium hydroxide solution. Shake and filter if necessary, then pass hydrogen sulfide into each solution. What has been the reaction in each case? Is each color change on heating the red lead accompanied by decomposition? Test this by heating another small portion till only the first change has occurred, then cooling.

b. Treat another small sample of red lead with dilute nitric acid, stirring till the change is complete. Filter and add sodium hydroxide solution to the filtrate, first in small amounts then

in excess, and account for the changes. What is the residue insoluble in nitric acid? Is it soluble in sodium hydroxide solution? In hydrochloric acid (hood)? What are the products of the reaction with hydrochloric acid? Warm to dissolve the lead chloride and test the solution by adding sodium hydroxide solution and compare with the reaction of the nitrate (the first filtrate, above).

104. Stannous and Stannic Tin.

a. Dissolve about 1 gram of tin in concentrated nitric acid diluted with an equal volume of water, filter and wash. What is the precipitate? Keep the filtrate and at the end of this experiment devise and apply a test to determine the presence or absence of tin. Dissolve a small portion of the precipitate in potassium hydroxide solution. Dissolve the rest in as little dilute hydrochloric acid as possible (A). What is formed in each case? Test small portions of each of these solutions by saturating with hydrogen sulfide, then acidify with hydrochloric acid and account for every change that takes place in each solution. Dilute a small portion of the hydrochloric acid solution (A) and add potassium permanganate solution (hydrochloric acid may reduce potassium permanganate solution if sufficiently concentrated). Is this stannous or stannic tin?

b. Dissolve 1 gram of tin by warming with concentrated hydrochloric acid diluted with an equal volume of water, avoiding an excess of the acid. Dilute and add potassium permanganate solution to a small portion. Account for the effect. Add potassium hydroxide solution to another portion till a permanent precipitate forms, which on addition of more potassium hydroxide and warming redissolves. What is in solution? Pass hydrogen sulfide into this alkaline solution and also into the acid solution. Compare with the same tests in *a* and account for the difference.

105. The Hydrogen Equivalent of Tin and Its Atomic Weight. Quantitative.

a. Repeat the manipulation described in Exp. 93 using about 0.1 to 0.15 gram of tin, and place in the test-tube a small piece

of platinum wire with which the tin may come in contact, to hasten its solution. Dissolve the tin in concentrated hydrochloric acid instead of the dilute, warming if necessary. Calculate the equivalent weight of tin, and by referring to Exp. 52 calculate the equivalent weight of tin from that experiment. Account for the difference.

b. Apply Dulong and Petit's Law to determine the atomic weight from these equivalents by determining the specific heat as follows. Take a piece of compact tin weighing 5 to 10 grams, or if a compact piece is not available melt the required amount of feathered or granulated tin in a crucible, and file down until a bright smooth surface is obtained. Weigh this to within 0.1 gram, place in a dry test-tube, and immerse the test-tube in a beaker of boiling water for ten to fifteen minutes, until the tin shall have reached the temperature of the water. Measure out in a small dry beaker 25 c.c. of water at the room temperature, and provide it with a stirrer made by flattening the end of a glass rod. Read the temperature of the water carefully, remove the tin from the boiling water and drop it directly from the test-tube into the beaker of water. Stir well, and after a moment or two read the temperature of the water again. If the temperature is read too soon error will result from incomplete distribution of the heat between the tin and the water, if too late, there may be an appreciable error introduced by loss of heat by radiation. Now read the temperature of the boiling water and from this data calculate the specific heat of the tin as follows: The change in temperature of the water times its volume measured in c.c. gives roughly the number of calories supplied by the tin (this is introducing a slight error by ignoring the heat capacity of the beaker and stirrer, and loss of heat by radiation), and that divided by the change in temperature of the tin times its weight gives the amount of heat supplied by 1 gram of the tin in changing its temperature by 1° , which is its specific heat. Apply Dulong and Petit's Law and give the true atomic weight of tin.

CHAPTER XXI

CHROMIUM

106. The Preparation of Sodium Chromate from Chrome Iron Ore.

Mix thoroughly 5 grams of sodium peroxide with 2 grams of well ground chrome iron ore and heat gently, keeping the mixture just melted for about twenty minutes. Cool and dissolve in a small amount of warm water and filter. What is the precipitate? Add hydrochloric acid to the filtrate as long as the orange color formed as the acid comes in contact with the solution changes back to the original yellow color on stirring. Explain these changes after the experiment is completed. What two salts are now present in this solution? Look up their solubilities and devise a method of taking advantage of their differences in solubility in hot and cold water, to accomplish their separation, and carry this out. Dissolve some of the crystals of sodium chromate and to one portion of the solution add silver nitrate solution, then acidify with nitric acid. To another portion add sulfuric acid and note the color change, then warm, adding alcohol drop by drop, and again note the color change. Compare these colors with those of chromates, dichromates and any chromium salt, and account for each change of color in the solution.

107. Chromyl Chloride.

Grind together 2 grams of potassium chromate and 1 gram of sodium chloride, place a small portion in a dry test-tube and add two or three drops of concentrated sulfuric acid slowly. Warm and collect a drop or two of the distillate which condenses on the walls of the test-tube by pouring it from the test-tube into a beaker containing water. Test this solution with litmus.

Add nitric acid and silver nitrate solution to one portion, to another portion add silver nitrate solution without acidifying. Explain the result in each case.

108. The Decomposition of Chromates by Heat.

a. Heat a small portion of well ground potassium dichromate in a dry, hard glass test-tube. Do the crystals contain water of crystallization? Is the substance volatile at this temperature? Heat till reaction ceases (how may that point be determined?), testing to determine what gas is evolved. Add water to the residue in the test-tube after cooling and heat if necessary to accomplish solution. Filter. What is the precipitate? Is it soluble in sulfuric acid? What is the filtrate?

b. To 5 c.c. of saturated potassium dichromate solution add concentrated sulfuric acid slowly till a permanent precipitate forms. What is the precipitate? Wet a piece of filter paper in this mixture and explain the color change. Dry the precipitate by decanting as much of the liquid as possible and absorbing the rest on a porous plate, and heat some of this dried material in a dry test-tube. Is it volatile? Does it decompose? Cool and treat the product with water and compare with the products obtained by heating potassium dichromate.

109. Chrome Alum.

Calculate the amount of sulfuric acid necessary to make chrome alum from 5 grams of potassium dichromate, reducing with alcohol. Dissolve 5 grams of powdered potassium dichromate in a small amount of water, add the calculated amount of sulfuric acid diluted with an equal volume of water, heat the solution and add 50 per cent alcohol, drop by drop, till reduction is complete. Set most of this solution aside to crystallize. To a small portion add sodium hydroxide solution, first in small quantities then in excess, and explain the changes. If chrome iron ore were fused with sodium hydroxide instead of the peroxide (Exp. 106) what would be the resulting compound?

CHAPTER XXII

MANGANESE

110. Compounds Showing the Varying Valence of Manganese.

a. Melt 4 grams of potassium hydroxide in an iron crucible and add gradually 2 grams of powdered manganese dioxide, increasing the heat as necessary to keep the mixture just melted, and stirring with an iron rod or file. In this procedure the manganese undergoes auto-oxidation, part being oxidized to hexavalent manganese while the other part is reduced to trivalent. Write the equation, assuming that the oxides are formed. Which of these oxides would be acidic, and what would be the effect of the potassium hydroxide on the acid oxide? Dissolve the fused mixture in distilled water and decant through a filter. Keep this filtrate for *d* and the insoluble residue for *b*.

b. Wash the residue with distilled water until the wash water is colorless. Some of the original manganese dioxide and some manganic oxide will be present in this residue. Add dilute sulfuric acid. Which oxide will dissolve? Allow to settle and decant the clear sulfuric acid solution from the residue, (save the residue for *c*), then make the solution alkaline with potassium hydroxide. What is formed? Keep this test for comparison later.

c. Wash the residue insoluble in sulfuric acid (what is it?) with distilled water till the wash water is colorless, then add dilute sulfuric acid, warm and add alcohol or sodium sulfite solution, drop by drop. What is the reaction? Make this solution alkaline with potassium hydroxide and compare with manganous hydroxide, made by adding potassium hydroxide to manganous sulfate, and with the manganic hydroxide prepared above. Which is it? Pour off as much of the supernatant liquid as possible and allow the precipitate to stand several

hours, and compare with the manganic hydroxide again. Account for the change.

d. Pour a part of the green filtrate from the fusion mixture into a large excess of water. What is formed? What is the oxidizing agent? The reducing agent? Acidify another portion with sulfuric acid and account for the change in color of the solution and for the precipitate formed. Add sodium sulfite solution to an alkaline and an acid solution of potassium permanganate, and to some of the green manganate solution. Which oxide of manganese is formed when alkaline solutions of manganates and permanganates are reduced? When an acid solution of permanganate (can an acid solution of a manganate be formed?) is reduced what is the valence of the manganese in the resulting compound? Devise and apply a test to prove your answer to these questions.

e. Add dilute nitric acid and some red lead to a solution of manganous sulfate, heat to boiling then allow the precipitate to settle and note and account for the color of the supernatant liquid.

CHAPTER XXIII

IRON

111. The Preparation of Ammonium Ferrous Sulfate.

Dissolve 5 grams of iron nails or wire by heating with the calculated amount of sulfuric acid diluted with four times its volume of water (hood) adding water if necessary to replace that lost by boiling, but keeping the final volume under 50 c.c. When the reaction has ceased filter, add a few drops of dilute sulfuric acid and the calculated amount of solid ammonium sulfate necessary for ferrous ammonium sulfate, heat to boiling (add water only if necessary to dissolve the ammonium sulfate at the boiling temperature) and as soon as solution is complete cool rapidly by holding under running water and stirring. Drain the crystals from the supernatant liquid, wash once with a small amount of cold distilled water, and dry the crystals on filter paper and finally by standing in the air. Weigh and calculate the per cent. yield (*i.e.* the percentage which the amount formed is of the theoretical amount obtainable from 5 grams of iron). Dissolve a small portion and test the solution with litmus. Save this preparation for Exps. 112 and 116.

112. Ammonium Iron Alum.

a. Dissolve 5 grams of the ferrous ammonium sulfate prepared in Exp. 111 in 15 c.c. of distilled water, heat to boiling and add concentrated nitric acid drop by drop with constant boiling until the addition of more nitric acid does not cause the formation of a dark brown color (to what is this color due?). Crystallize the alum by cooling and evaporation, and remove the crystals from the liquid, dry on filter paper and place in a stoppered bottle for use in Exp. 113. Dissolve a small portion and test the solution with litmus. Compare with the ferrous salt.

b. Test solutions of ferrous and ferric salts with ammonium hydroxide, potassium permanganate solution and potassium iodide solution. Contrast the effects of the two iron salts in each case. Write equations for each reaction.

113. Determination of the Water of Crystallization and of Ferric Oxide in Ammonium Iron Alum. Quantitative.

Weigh a clean dry porcelain crucible, place in it 0.2 to 0.5 gram of the ammonium iron alum prepared in Exp. 112 (if these crystals have become opaque, or formed a brown deposit on the surface they will not give good results as decomposition has already started) and determine the water of crystallization according to the method used for aluminium alum, Exp. 100. After constant weight has been attained at the low temperature required for driving off the water, heat the crucible slowly to red heat with the direct flame, cool and weigh, and repeat till it comes to constant weight. The residue left in the crucible is ferric oxide. From the weights of the water, the ferric oxide and the original weight of the alum taken calculate the per cent. of water and iron in the alum.

114. The Hydrolysis of Ferric Salts, and Colloidal Ferric Hydroxide.

a. Heat a small crystal of hydrated ferric chloride in a dry, soft-glass test-tube, testing the gas evolved with moist litmus paper. What is evolved? Heat till the test-tube is entirely dry, cool, add water, filter and test the filtrate for iron. What is the insoluble residue?

b. Make 50 c.c. of a 5 per cent. solution of ferric chloride, add sodium carbonate solution as long as the precipitate first formed redissolves (explain the phenomena observed here) and if a permanent precipitate forms add dilute hydrochloric acid till it dissolves. Dialyse in a collodion sack (Exp. 71 b) changing the water frequently at first and test the dialysate for chlorides. When the dialysate shows only traces of chlorides after standing in contact with the sack for about an hour remove the sack and

test the solution of colloidal iron for chlorides. Add a concentrated solution of sodium chloride to this solution. Is this a physical or a chemical change? Add a dilute acid to another portion of the colloidal iron hydroxide solution and account for the change in color.

c. Recall the effect of ferrous and ferric sulfate solutions on litmus. In which valence does iron show most marked metallic properties?

115. Cast Iron and Steel.

Dissolve small samples of cast iron and steel in dilute hydrochloric acid, testing the gases evolved for hydrogen sulfide. Note the amount and appearance of the residue in each case. Filter and mix the dried residues with copper oxide and heat each separately in a hard glass test-tube, testing the evolved gas for carbon dioxide. Dissolve about a gram of steel filings by stirring with potassium cupric chloride solution (what is the white precipitate that forms and redissolves?). Note the appearance of the residue and test the gases evolved when it is heated with copper oxide. What elements besides iron are present in cast iron and steel?

116. The Oxidation of Ferrous Ammonium Sulfate by Potassium Permanganate and by Potassium Dichromate. Quantitative.

Make 0.1 formalar solutions of potassium permanganate and potassium dichromate by weighing accurately the amount required for 100 c.c., placing this amount in the measuring cylinder, dissolving in distilled water, then adding water to make just 100 c.c. (be careful that none of the solution is lost). *The rest of this experiment must be performed in one laboratory period.* Make 100 c.c. of a 0.1 formalar ammonium ferrous sulfate solution, weighing out the salt prepared in Exp. 111 accurately (remember that these crystals contain water) and place exactly 50 c.c. of this solution in each of two beakers. Add to each solution 5 c.c. of dilute sulfuric acid, heat to boiling and to the first beaker add the prepared solution of potassium

permanganate from a burette until the purple color of the permanganate just becomes permanent, and record the amount of permanganate solution required. To the second beaker containing ammonium ferrous sulfate heated to boiling add potassium dichromate solution from a burette, 1 c.c. at a time, testing a drop of the solution after each addition by bringing it in contact with a drop of potassium ferricyanide solution on a white dish or test plate. As long as a blue precipitate or solution is formed on mixing these drops there is still some unchanged ferrous sulfate in the solution, and potassium dichromate solution should be added only until the test drop no longer gives a blue color. Record the amount of potassium dichromate solution required. How do these two oxidizing agents compare in oxidizing power, or in the amount of oxidation accomplished by equal molecular quantities of each of the salts? In order to make a normal solution of each so that the oxygen available for oxidations should be equivalent to 1 gram of hydrogen per liter, how much of each salt would be required for a liter of solution?

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